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Insoluble Coatings for Stirling Engine Heat Pipe Condenser Surfaces

Phase II: Final Report

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PROJECT SUMMARY PAGE

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SUMMARY:

This report describes the work performed by Thermacore, Inc., Lancaster, Pennsylvania for a Phase II SBIR program under NASA Contract No. NAS3-26925, entitled *Insoluble Coatings for Stirling Engine Heat Pipe Condenser Surfaces*. The work was performed between June 1993 and March 1996.

Thermal energy sources for Stirling engines typically have non-uniform temperatures and heat fluxes. For solar and nuclear applications, liquid metal heat pipe receivers can be used to deliver this thermal energy at a uniformly high temperature to the Stirling engine heater heads. The use of heat pipe receivers greatly enhances system efficiency and potential life span. One issue raised during the heat pipe receiver design phase is the potential solubility corrosion of the heater head section by the liquid metal working fluid. To address this issue, the Phase I program studied nickel aluminide coatings for nickel-based superalloys and developed and demonstrated a vehicle to test candidate materials and coatings. The Phase II program continued this work effort, initiated a study of nickel aluminide coatings for capillary wick structures, and fabricated a coated full-scale, partial segment of the current Stirling engine heat pipe design for the Stirling Space Power Convertor (SSPC) program.

In Phase II, uncoated and nickel aluminide coated Nickel 200 (Ni200), Inconel 718 (In718) and Udimet 720LI (Ud720LI) sample tubes were tested in condensing sodium at 1073K and a condensation heat flux of 25W/cm². Testing of the Ni200 and Ud720LI samples tubes was stopped at 2500 and 1886 hours, respectively, because of irreparable test vehicle leaks. Analyses of the samples indicated an average corrosion rate of approximately 0.0033cm/yr for the uncoated Ni200 tubes; mixed results for the nickel aluminide coated Ni200 tubes; and no sign of corrosion for the uncoated and nickel aluminide coated Ud720LI tubes. Testing of the In718 sample tubes was stopped at 8767 hours (contract goal). Analyses of these samples indicated essentially no sign of corrosion for both the uncoated and nickel aluminide coated In718 tubes. In each test vehicle, two sample tubes (one uncoated, one coated) were covered with uncoated, Stainless Steel 316 screen (SS316), and two sample tubes (one uncoated, one coated) were covered with uncoated, Ni200 screen. Analyses of the samples showed little to no sign of corrosion for any of the SS316 screens; moderate corrosion for the Ni200 screen in the Ni200 and Ud720LI test vehicles; and, severe deterioration of the Ni200 screen in the In718 test vehicle.

In addition, the Phase II effort successfully demonstrated a two-step nickel aluminide coating process for groove wick structures and interior wall surfaces in contact with liquid metals; demonstrated a one-step nickel aluminide coating process for Ni200 screen wick structures; and developed and demonstrated a two-step aluminum-to-nickel aluminide coating process for Ni200 screen wick structures.

In Phase II, four durability heat pipes were used to test the coatings and processes developed. The first heat pipe incorporated the two-step nickel aluminide coating for groove wick structures and interior surfaces. The second heat pipe incorporated the two-step nickel aluminide coating for the interior surfaces and the one-step nickel aluminide coating for Ni200 screen wick structures. The third heat pipe incorporated the two-step nickel aluminide coating for the interior surfaces and the two-step aluminum-to-nickel aluminide coating for Ni200 screen wick structures. The fourth heat pipe had no coating on the interior wall surfaces or the Ni200 screen wick structures. Each of the heat pipes was operated for 5000 hours at 1073K and at a condensation heat flux of 25W/cm². Analyses of all post-test samples indicated no sign of corrosion for the wall surfaces and groove wick structures with the two-step nickel aluminide coating; no sign of corrosion for the screen wick structures with the one-step nickel aluminide coating; no sign of corrosion for the screen wick structures with the two-step aluminum-to-nickel aluminide coating; no sign of corrosion for the uncoated wall surfaces; and moderate corrosion for the uncoated screen wick structures.

As an example of a real world application, a full-scale, partial segment of the current Stirling Space Power Convertor heat pipe was fabricated and coated using the best application processes developed earlier in the program. The inner surfaces of the heat pipe were coated using the two-step nickel aluminide coating process, and the screen wick structure was coated using the two-step aluminum-to-nickel aluminide coating process. At the completion of the program and using Thermacore IR&D funding, the heat pipe was charged with high purity sodium, processed to remove non-condensable gases, and is scheduled to be life tested for up to ten years as a Phase III effort.

SUBMITTED: _____

Pat M. Dismore
Principal Investigator Signature

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EXECUTIVE SUMMARY

Thermacore, Inc., Lancaster, Pennsylvania has completed a Phase II SBIR program under NASA Contract No. NAS3-26925, entitled *Insoluble Coatings for Stirling Engine Heat Pipe Condenser Surfaces*, to develop and demonstrate a practically insoluble coating for nickel-based superalloys for high temperature sodium heat pipe applications. The program was monitored by NASA Lewis Research Center, Cleveland, Ohio. The work was performed between June 14, 1993 and March 31, 1996.

Stirling heat engines are being developed for electrical power generation on manned and unmanned earth orbital and planetary missions and also for terrestrial applications for utility grid and remote power generation. Dish Stirling solar systems and nuclear reactor Stirling systems are two promising applications of Stirling engine technology. Sources of thermal energy used to drive the Stirling engine typically have non-uniform temperatures and heat fluxes. Liquid metal heat pipe receivers are often used as heat transformers to uniformly deliver thermal energy at high temperatures to the heater heads of these Stirling engines. The use of heat pipe receivers greatly enhances system efficiency and potential life span.

One issue raised during the design phase of heat pipe receivers is the potential solubility corrosion of the heater head section by the liquid metal working fluid. For Stirling engine heat pipe applications, the Phase I program initiated a study of nickel aluminide coatings for nickel-based superalloys and developed and demonstrated a vehicle to test candidate materials and coatings. The principal objective of the Phase II program was to develop and demonstrate a practically insoluble coating for nickel-based superalloys for Stirling engine heat pipe applications. For all practical purposes, the coating should eliminate the potentially life limiting solubility corrosion that occurs in sodium heat pipes. Specific technical objectives of this program were:

- Determine the solubility corrosion rates for Nickel 200 (Ni200), Inconel 718 (In718) and Udimet 720LI (Ud720LI) in a simulated Stirling engine heat pipe environment,
- Develop coating processes and techniques for capillary groove and screen wick structures,
- Evaluate the durability and solubility corrosion rates for capillary groove and screen wick structures coated with an insoluble coating in cylindrical heat pipes operating under Stirling engine conditions,
- Design and fabricate a coated full-scale, partial segment of the current Stirling engine heat pipe for the Stirling Space Power Converter (SSPC) program.

In Phase II, Thermacore and NASA Lewis Research Center reviewed and updated the requirements for a typical space Stirling engine heat pipe receiver. In addition, a letter questionnaire was distributed to research personnel in the coatings, corrosion protection, Stirling engines, and high temperature heat pipe fields. The purpose of the questionnaire was to gather information related to the interactions between alkali liquid metals and nickel-based superalloy substrates and to ascertain experiences with coatings which prevent or reduce the interaction of the liquid metals and the nickel-based superalloys. During the program, Thermacore distributed forty-one letter questionnaires and received seven responses. A copy of this final report will be provided to the individuals who responded to the questionnaire.

To study solubility corrosion of nickel-based alloys in sodium, uncoated and nickel aluminide coated Ni200, In718 and Ud720LI sample tubes were tested in condensing sodium at 1073K and at a condensation heat flux of 25W/cm². Testing of the Ni200 and Ud720LI sample tubes was stopped at 2500 and 1886 hours, respectively, due to sodium leaks in the test vehicles. The analyses of these samples indicated an average corrosion rate of approximately 0.0033cm/yr for the uncoated Ni200 tubes; mixed results with a large variation in corrosion rates for the nickel aluminide coated Ni200 tubes (average corrosion rate of approximately 0.0044cm/yr); and no sign of corrosion for the uncoated and nickel aluminide coated Ud720LI tubes. Testing of the In718 sample tubes was stopped at 8767 hours (contract goal, 8760 hours). Analyses of these samples indicated essentially no signs of corrosion for the uncoated and nickel aluminide coated In718 tubes.

In each test vehicle, two sample tubes (one uncoated, one coated) were covered with two layers of uncoated, 100 mesh Stainless Steel 316 (SS316) screen, and two sample tubes (one uncoated, one coated) were covered with two layers of uncoated, 100 mesh Ni200 screen. Analyses of the samples for all of the test vehicles indicated little to no sign of corrosion (only one of eighteen samples showed corrosion) for the SS316 screens. Analyses of the samples for the Ni200 and Ud720LI test vehicles indicated moderate corrosion for the Ni200 screens. In the In718 test vehicle, the Ni200 screens were severely deteriorated.

In addition, the Phase II effort successfully demonstrated a two-step nickel aluminide coating process for capillary groove wick structures in contact with liquid metals; demonstrated a one-step nickel aluminide coating process for Ni200 screen wick structures; and developed and demonstrated a two-step aluminum-to-nickel aluminide coating process for Ni200 screen wick structures. Hitemco's two-step, pack cementation, nickel aluminide coating was applied uniformly and consistently to In718 groove wick structures. After 5000 hours of testing, the coating showed no sign of corrosion in condensing sodium at 1073K and at a condensation heat flux of 25 W/cm². Hitemco's one-step, pack cementation, nickel aluminide coating was applied uniformly and consistently to Ni200 screen wick structures. After 5000 hours of testing, the coating showed no sign of corrosion in condensing sodium at 1073K and at a condensation heat flux of 25 W/cm². The two-step aluminum (vapor deposited by Titanium Finishing Company)-to-nickel aluminide coating process was applied uniformly and consistently to Ni200 screen wick structures. After 5000 hours of testing, the coating showed no sign of corrosion in condensing sodium at 1073K and at a condensation heat flux of 25 W/cm². Aluminum powder doped sodium test pipes did not produce a uniform nickel aluminide coating on wall surfaces, groove wick structures or screen wick structures. As a result, this process does not appear to be feasible for coating heat pipe wick structures without substantial further development.

The results of this program indicate that the corrosion rate for uncoated nickel-based superalloys such as In718 and Ud720LI appear to be nearly negligible in condensing sodium at 1073K and 25 W/m² condensation heat flux rates. However, it may be beneficial to coat with nickel aluminide systems that are required to last for tens of years and systems that have thin cross-sections. If a coating is desired, the two-step nickel aluminide coating process is recommended for interior wall surfaces and relatively large groove wick structures. For screen wick structures that require ductility during installation and spot welding, the two-step aluminum-to-nickel aluminide coating process is recommended. The screen is installed in the aluminum coated state and, after installation, is converted to nickel aluminide. A one-step nickel aluminide coating process is also available for screen wicks that do not require ductility during installation (e.g., rolled and stuffed into a straight cylindrical heat pipe of moderate diameter).

As an example of a real world application, a full-scale, partial segment of the current SSPC heat pipe was fabricated and coated using the best processes developed in the program. The inner surfaces of the heat pipe were coated using the two-step nickel aluminide coating process, and the screen wick structure was coated using the two-step aluminum-to-nickel aluminide coating process. After fabrication and using Thermacore IR&D funding, the heat pipe was charged with high purity sodium, processed to remove non-condensable gases, and is scheduled to be life tested for up to ten years as a Phase III effort.

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1.0 INTRODUCTION

Stirling heat engines are being developed for use on manned and unmanned earth orbital and planetary surface missions for electrical power generation in space and also for terrestrial applications for utility grid and remote power generation. Liquid metal heat pipe receivers are often used as heat transformers or isothermalizers to deliver thermal energy at uniformly high temperatures to the heater head (heat input section) of these Stirling engines. Through analysis and prototypes, heat pipe receivers have been shown to maximize system efficiency and significantly increase the durability of the heater head. The thermal energy can be supplied by solar concentrators, pumped liquid metal loops in nuclear reactors, or combustion of solid, liquid or gaseous fuels. Two examples of liquid metal heat pipe receiver / Stirling engine generators are the Dish Stirling system and the nuclear reactor / Stirling engine system.

Dish Stirling systems are one of the most promising applications of Stirling engine technology. In these systems, solar energy is concentrated by a parabolic reflector and directed to the heater head. Typically, the Stirling heater consists of small diameter tubes that contain the working fluid (e.g., helium or hydrogen) as it moves between the expansion space and the regenerator. Optimum engine efficiency results when the engine hot end and heater head tubes operate at a uniform temperature. The design hot-end temperature for a Stirling engine is typically set in the range of 823-1073K. Liquid metal heat pipe receivers can be used to uniformly heat the engine hot end and heater head tubes. Microgravity applications can also use a variation of this receiver design. Dish Stirling systems are described in Reference [1].

As part of the SP-100 program, nuclear reactor / Stirling engine systems were being developed for electrical power generation in space. Typically, the nuclear reactor is cooled with a liquid metal pumped loop thermally coupled to the Stirling engine by a liquid metal heat pipe receiver. The starfish heat input section of this receiver design consists of metal fins with internal passageways for the working fluid. As mentioned before, optimum engine efficiency results when the engine hot end and heat input fins operate at a uniform temperature. Again, liquid metal heat pipes can be used to uniformly heat the engine hot end and heat input fins. The Stirling Space Power Convertor project and the starfish heater / heat pipe are described in References [2] and [3].

Heat pipes are sealed evacuated devices which transfer heat by evaporating and condensing a working fluid. In addition, heat pipes are passive devices which do not require external power for operation. Typical liquid metal heat pipes operating in the 823-1073K temperature range use sodium metal as the working fluid. For the Stirling electrical power generation application, the heat input into the evaporator section causes some of the working fluid to vaporize. As a result, the local vapor pressure in the evaporator section increases above the pressure in the condenser section. The pressure differential causes the vapor to flow to the condenser section where it condenses. In the Stirling engine applications, the condensation occurs on the heater head tube or fin surfaces. This process delivers the thermal energy to these surfaces at a uniform temperature. The condensed fluid returns to the evaporator section by gravity and/or capillary pressure developed in the wick structure.

Stainless steels and nickel-based superalloys are standard materials of construction for high temperature heat pipes and heater heads for Stirling engines operating in the 823-1073K temperature range. At these operating temperatures, some components of these materials are appreciably soluble in working fluids such as sodium, potassium and NaK. Over a typical mission life span of seven to ten years, essentially pure working fluid condensate will condense on the heater head surfaces. The condensate will leach the soluble components of the heater head material and transport them to the evaporator section of the heat pipe

receiver. When the working fluid is again evaporated, the soluble materials are precipitated and essentially pure working fluid is returned to the condenser section to leach more material from the tube or fin surfaces. Since the condensation heat fluxes are relatively high, this continuous solubility cycle which corrodes the heater head surfaces is a particularly important issue for Stirling engine heat pipe receivers. In this application, condensation heat fluxes of 20-25W/cm² are common. At 25W/cm², approximately 760,000 liters of sodium per year will condense on a heater head for a 33% efficient 25kW_e Stirling engine.

Solubility corrosion has the potential to contribute to a number of premature failure mechanisms. For sodium at 993K, pumped loop solubility corrosion rates for nickel based superalloys are typically 0.002-0.013cm/yr^[4]. At these rates, the wall thickness of a heater head tube or fin could be reduced by 0.025-0.127cm over ten years. In some cases, this corrosion depth is greater than the starting wall thickness and in most cases is at least 50% of the starting thickness. Another potential failure mechanism is plugging of the evaporator wick structure with the precipitated heater head material. Typical heat pipe wick structures have pore sizes of approximately 1x10⁻⁵m. A reduced wick porosity can cause high liquid flow pressure drops that exceed the wick pumping capability. The potential result is a dryout situation and local overheating of the receiver envelope.

Solubility of nickel-based superalloy components, preferential corrosion of grain boundaries, and the effects of excess oxygen in the working fluid are additional mechanisms for potential corrosion of the heater head materials. Evidence also indicates that the synergistic effect of two or more of these mechanisms may further increase the rate of corrosion. Since heat pipe environments are sometimes more severe than pumped loop environments, these observations emphasize the need for actual heat pipe tests to establish baseline solubility corrosion data for heat pipe receiver materials under simulated Stirling engine conditions. However, the pumped loop data provides sufficient, independent evidence that a practically insoluble coating for heater head surfaces may be required to meet the life and reliability requirements of the Stirling engine system missions.

For liquid metal nuclear reactor systems, the metallurgical coatings developed for wear and surface damage resistance for nickel-based superalloys have also been shown to greatly reduce solubility corrosion. In particular, nickel aluminide diffusion coatings have been tested in sodium at 948K and have demonstrated the potential to reduce corrosion rates to approximately 0.00008cm/yr (100 times less than the average for uncoated nickel-based superalloys)^[5]. This coating and others have the potential to reduce corrosion rates to near negligible levels, essentially eliminate the effects of preferential grain boundary attack, and minimize the need for ultra pure working fluids. However, before these coatings gain acceptance for Stirling engine heat pipe receiver applications, coating techniques for the wicking must be developed and a series of heat pipe tests completed to demonstrate the performance of the coatings under simulated Stirling engine conditions.

Under the Phase I program (NASA Contractor Report 191188), uncoated and nickel aluminide coated Nickel 200 tubes were tested for 1000 hours at 1073K and a condensation heat flux of 25W/cm² in a simulated Stirling engine environment (solubility corrosion test vehicle). Chromalloy coated the Nickel 200 tubes with a two-step nickel aluminide application process. Analyses of the tube samples showed no visible signs of corrosion of either the uncoated or coated samples. The results indicated that the heat pipe environment was not directly comparable to liquid metal pumped loop data, that nickel aluminide was still a leading candidate for solubility corrosion protection, and that longer duration tests were recommended to definitely conclude whether coatings are required.

The principal objective of this Phase II program was to develop and demonstrate a practically insoluble coating for nickel-based superalloys for Stirling engine heat pipe applications. For all practical purposes, the coating should eliminate the potentially life limiting solubility corrosion that occurs in sodium heat pipes. Specific technical objectives of this program were:

- Determine the solubility corrosion rates for Nickel 200, Inconel 718 and Udimet 720LI in a simulated Stirling engine heat pipe environment,
- Develop coating processes and techniques for capillary groove and screen wick structures,
- Evaluate the durability and solubility corrosion rates for capillary groove and screen wick structures coated with an insoluble coating in cylindrical heat pipes operating under Stirling engine conditions,
- Design and fabricate a coated full-scale, partial segment of the current Stirling engine heat pipe for the Stirling Space Power Converter program.

2.0 DISCUSSION

The Phase II SBIR program consisted of five technical tasks plus reporting. The first task combined two specific technical objectives. The first objective was to update the Phase I SBIR Stirling engine heat transport system requirements to meet the needs of current Stirling power conversion systems. The second objective was to prepare a letter questionnaire which was distributed to research personnel in the coatings, corrosion protection, Stirling engines, and high temperature heat pipe fields. The purpose of this questionnaire was to gather information related to interactions between alkali liquid metals and nickel-based superalloy substrates and to identify coatings which prevent or reduce these interactions. The second task was to perform solubility corrosion tests on materials which were candidates for space Stirling engine heat pipes or served as baseline references for the data being obtained. Under this task, three test vehicles were fabricated and operated to evaluate uncoated and nickel aluminide coated Nickel 200, Inconel 718 and Udimet 720LI materials. The objective of the third task was to study the application of nickel aluminide coatings to heat pipe wick structures, namely capillary grooves and screens. The fourth task was to determine the durability and suitability of nickel aluminide coatings under heat pipe operating conditions. The objective of the fifth task was to design and fabricate a heat pipe which represents a full-scale, partial segment of the heat transport system in the Stirling Space Power Convertor program. This heat pipe was fabricated from Inconel 718 and used a nickel aluminide coated screen wick structure. The following sections detail the work effort completed during the Phase II program.

Table 1. Typical Stirling Engine Heat Pipe Receiver Requirements.

Parameter	Magnitude
Operating Temperature	1023-1073K
Working Fluid	High Purity Sodium (≈ 10 ppm oxygen)
Condensation Heat Flux	25 W/cm ²
Heater Head Geometries	Tubes or Fins
Heater Head Material	Inconel 718 or Udimet 720

2.1 TASK 1: SURVEY OF DESIGN REQUIREMENTS, CURRENT TECHNOLOGY, AND GOALS

The first objective of Task 1 was to update the Stirling engine heat transport section requirements established in Phase I to meet the needs of current Stirling power conversion systems. During the "kick-off" meeting held at NASA LeRC on June 23, 1993, the requirements for a typical space Stirling engine heat pipe receiver were reviewed. Table 1 shows the typical space Stirling engine heat pipe requirements. The requirements for the Phase II test vehicles were selected based on the requirements in Table 1 and discussions with NASA LeRC. Table 2 shows the requirements for the Phase II solubility corrosion test vehicles.

The second objective of Task 1 was to prepare a letter questionnaire which was distributed to research personnel in the coatings, corrosion protection, Stirling engines, and high temperature heat pipe fields. The purpose of this questionnaire was to gather information related to the interactions between alkali

Table 2. Requirements for Phase II Solubility Corrosion Test Vehicles.

Parameter	Magnitude
Operating Temperature	1073K
Operating Life	Up to 8760 Hours
Working Fluid	High Purity Sodium (10-20ppm Oxygen)
Condensation Heat Flux	25 W/cm ²
Condensation Surface	Tubes: 2.512 cm Nominal Diameter, 10.2 cm Length
Number of Test Vehicles	3 (One for each substrate material)
Condenser Substrate Materials	Nickel 200; Inconel 718; and Udimet 720LI
Number of Sample Tubes per Test Vehicle	6 (3 nickel aluminide coated and 3 uncoated)

liquid metals and nickel-based superalloy substrates and to ascertain experiences with coatings which prevent or reduce the interaction of the liquid metals and the nickel-based superalloys. Throughout the program, Thermacore distributed forty-one letter questionnaires. From these, seven responses were received; several responses are summarized below. At the end of the program, a copy of this final report was sent to the individuals who responded to the questionnaire.

In May 1994, Mr. James Moreno and Mr. Charles Andraka of Sandia National Laboratories responded by telephone to the questionnaire. At the time, their area of research involved studying small Haynes 230 (Ha230) reflux boilers with Inconel 600 (In600) thermowells and sodium-potassium alloy 78 (NaK-78) working fluid. The boiler survived 7500 hours of testing at 1023K. The test was ended when an In600 thermowell developed a leak. The In600 was attacked by the NaK at the grain boundaries. However, the Ha230 had only a few microns of surface regression^[6,7].

Dr. Nate Hoffman of Energy Technology Engineering Center (ETEC) responded by telephone to the letter questionnaire. In addition to compiling a bibliography of seven reports which discuss aluminide coatings and sodium corrosion^[8-14], he indicated that

- Essentially no corrosion of nickel aluminide coatings occurs in sodium environments in tests over many years,
- Materials which have corroded in other parts of test systems have not precipitated onto nickel aluminide coatings,
- Pure sodium is less corrosive than sodium saturated with nickel,
- Nickel aluminide coatings are frequently used in liquid metal fast breeder reactors for corrosion resistance, and

- Materials should contain at least 12% nickel to be successfully coated with nickel aluminide.

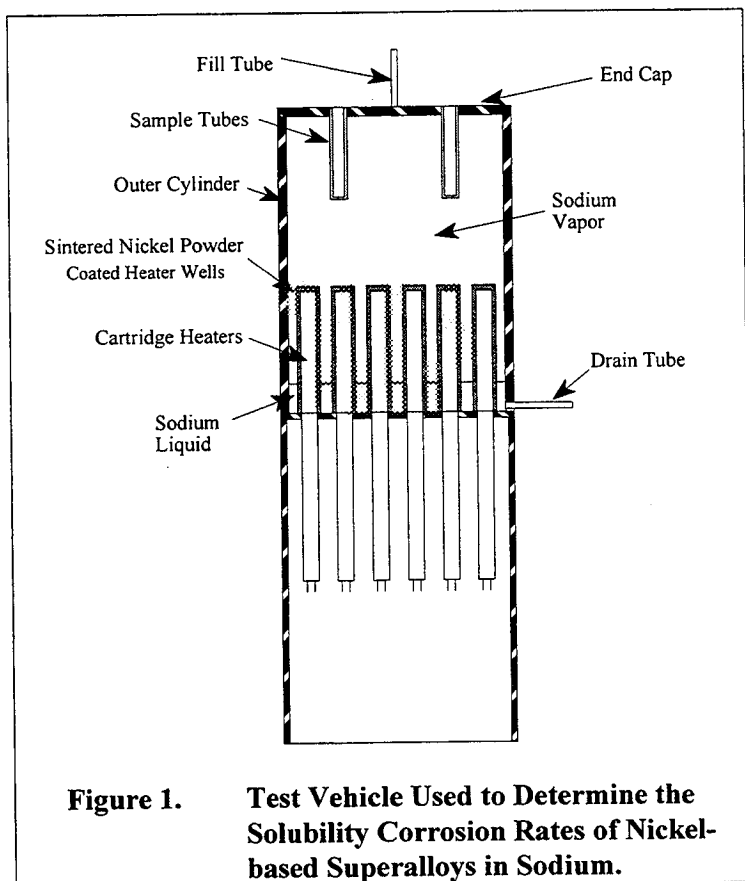
2.2 TASK 2: GENERATION OF A SOLUBILITY CORROSION DATA BASE FOR CANDIDATE MATERIALS FOR STIRLING ENGINE HEAT PIPE CONDENSERS

The objective of Task 2 was to perform solubility corrosion tests on materials which were candidates for space Stirling engine heat pipes or served as baseline references for the data being obtained. The solubility corrosion tests subjected the candidate materials to condensate generated in a sodium heat pipe. The goal of these tests was to establish a solubility corrosion data base for candidate materials. The development of the technology required to fabricate and operate the solubility corrosion test vehicles is discussed in Sections 2.2.1 through 2.2.4. Test results are given in Section 2.2.1. Figures 1 and 2 show typical solubility corrosion test vehicles. Figure 3 shows the control panel used to power and monitor the test vehicle.

2.2.1 Solubility Corrosion Test Vehicles

During the "kick-off" meeting at NASA LeRC on June 23, 1993, Thermacore and NASA personnel agreed on the fabrication of three solubility corrosion test vehicles. These test vehicles were used to perform solubility corrosion tests on three candidate materials for use in Stirling engine heat pipes. The materials selected for testing were Nickel 200 (Ni200), Inconel 718 (In718) and Udimet 720LI (Ud720LI). Table 3 shows the solubility corrosion test matrix.

Each solubility corrosion test vehicle was dedicated to six sample tubes of a single candidate material. Of the six tubes, three were coated with nickel aluminide and three were uncoated. Hitemco (Old Bethpage, NY) coated the sample tubes using their two-step nickel aluminide application process. For Ni200 tubes, the first step was a four hour, 1023K pack cementation process. Hitemco stated that the nickel aluminide coating forms more rapidly on pure nickel than on nickel alloys. As a result, a higher first step process temperature was required to form the nickel aluminide coating on the In718 and Ud720LI tubes. For these tubes, the first step was a four hour, 1193K pack cementation process.



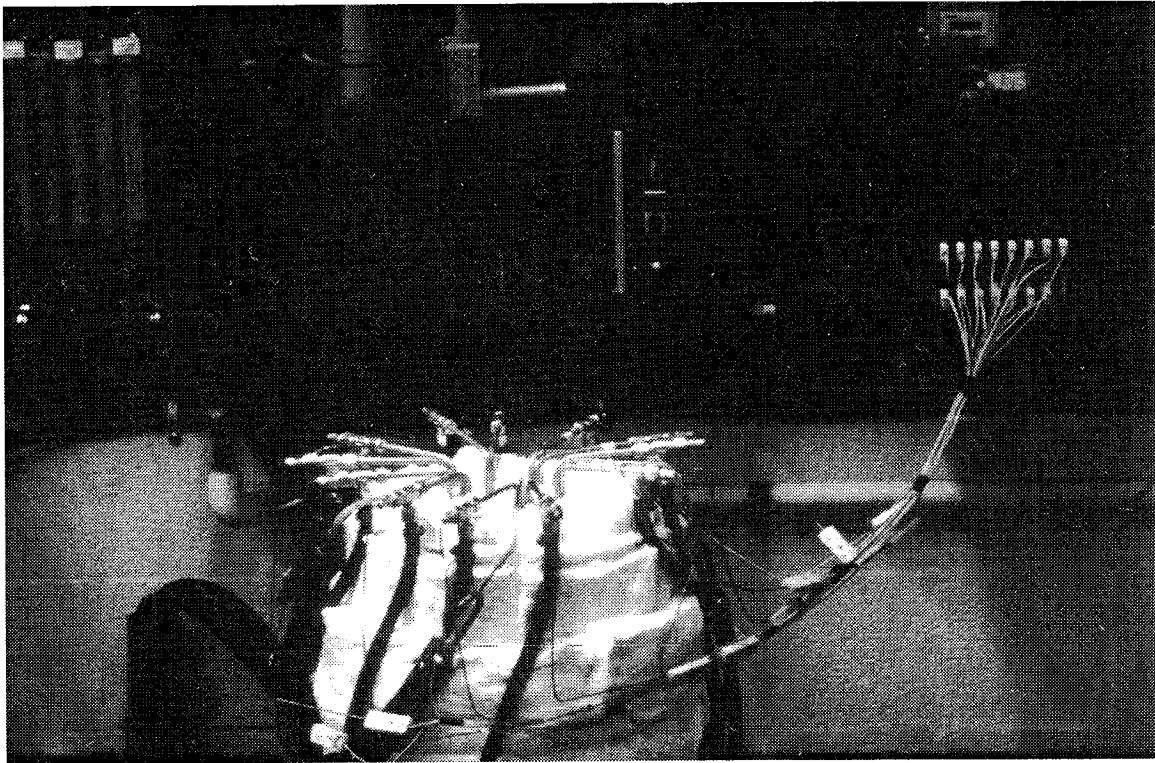


Figure 2. Insulated Solubility Corrosion Test Vehicle for Ni200 Sample Tubes.

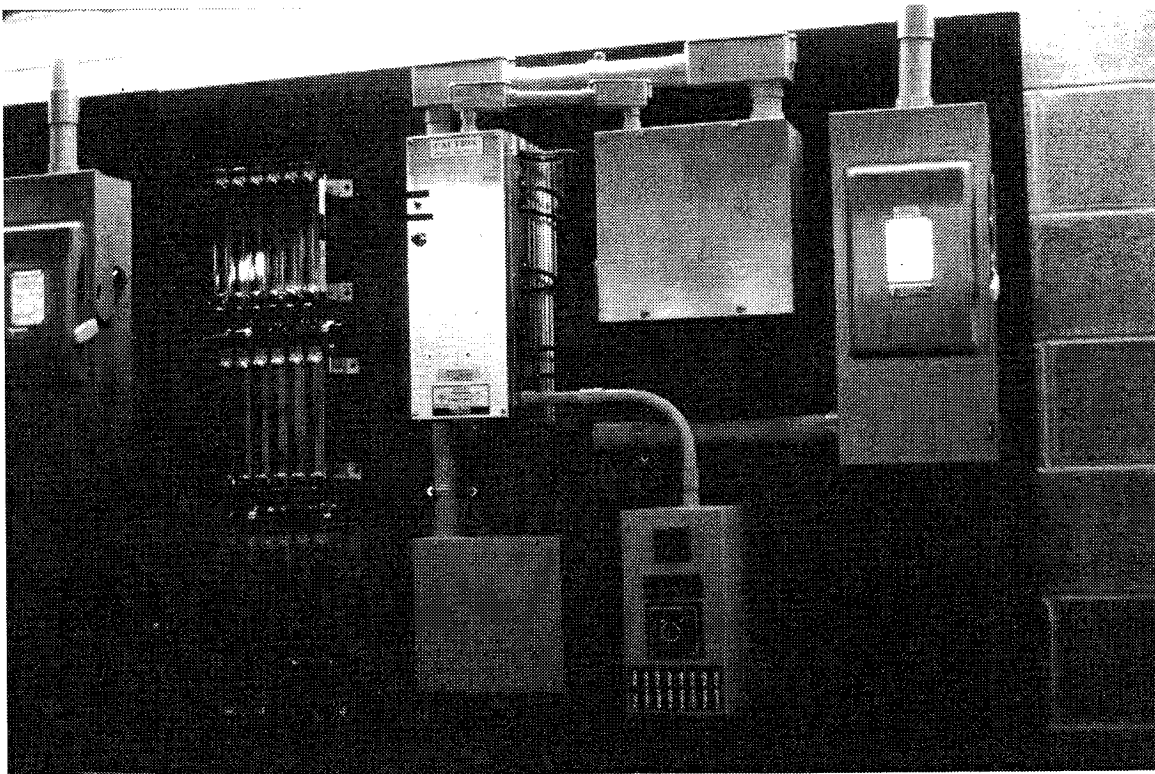


Figure 3. Control Panel for the Solubility Corrosion Test Vehicles

Table 3. Solubility Corrosion Test Vehicle and Reference Stripe Test Matrix.

Test Vehicle Number	Sample Tube Material	Coating Material	Screen Material	Reference Stripe 1 Material	Reference Stripe 2 Material	Reference Stripe 3 Material	Reference Stripe 4 Material
1	Nickel 200	Nickel Aluminide	None	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten
	Nickel 200	Nickel Aluminide	100 Mesh SS316				
	Nickel 200	Nickel Aluminide	100 Mesh Ni200				
	Nickel 200	None	None	Plasma-Sprayed Tungsten	Electroless Nickel	Plasma-Sprayed Tungsten	Electroless Nickel
	Nickel 200	None	100 Mesh SS316				
	Nickel 200	None	100 Mesh Ni200				
2	Inconel 718	Nickel Aluminide	None	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten
	Inconel 718	Nickel Aluminide	100 Mesh SS316				
	Inconel 718	Nickel Aluminide	100 Mesh Ni200				
	Inconel 718	None	None	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten
	Inconel 718	None	100 Mesh SS316				
	Inconel 718	None	100 Mesh Ni200				
3	Udimet 720LI	Nickel Aluminide	None	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten
	Udimet 720LI	Nickel Aluminide	100 Mesh SS316				
	Udimet 720LI	Nickel Aluminide	100 Mesh Ni200				
	Udimet 720LI	None	None	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten	Plasma-Sprayed Tungsten
	Udimet 720LI	None	100 Mesh SS316				
	Udimet 720LI	None	100 Mesh Ni200				

During the first step, an essentially pure layer of aluminum was applied to the sample tubes. The second step is a two hour, 1313K diffusion heat treatment. As a result, a coating thickness of approximately 0.008cm was applied to all sample tubes.

Two methods were used to determine the corrosion, if any, for the sample tubes. The first method was to measure the diameter of the uncoated and coated sample tubes before and after testing. The second method used reference stripes to protect the surface of the tube and to provide a reference line from which to measure corrosion. Photomicrographs were taken at the edge and center of the reference stripes and at the uncoated and nickel aluminide coated tube surfaces. The stripe edge photomicrographs were used to determine the corrosion rates in Table 7.

After initial machining, the outer tube diameters of nine Ni200, eight In718 and nine Ud720LI uncoated tubes were measured using the procedure discussed in Appendix A. Lines scribed on the inner diameter of each sample tube corresponded to surfaces on the outer diameters from which measurements were obtained. Hitemco then coated five Ni200, four In718 and five Ud720LI sample tubes with nickel aluminide to a thickness of approximately 0.0008cm. After coating, these tubes were centerless ground to a diameter of 2.515 cm. Again using the sample tube measurement procedure, Thermacore measured the outer diameters of the coated tubes. The uncoated and coated sample tube measurements are included as Appendix B. To help assure accurate measurements, all equipment for measuring the sample tubes was not employed for any other use between the initial and final measurements of each sample tube.

Each tube was prepared with four equally spaced reference stripes to provide protected surface contact lines from which to scale photomicrographs. On the uncoated and nickel aluminide coated In718 and Ud720LI sample tubes, the four reference stripes were plasma-sprayed tungsten. On the nickel aluminide coated Ni200 sample tubes, the four reference stripes were also plasma-sprayed tungsten. On the uncoated Ni200 sample tubes, two reference stripes were plasma-sprayed tungsten and two were electroless plated nickel. The plasma-sprayed tungsten was selected based on the results of the diffusion and thermal cycling tests discussed in the next section. The electroless plated nickel was selected based on its Phase I use and because it would not significantly diffuse into the Ni200 tube. The reference stripes were 0.013cm thick, 0.64cm wide and 9.53cm long. Table 3 shows the reference stripe materials which were applied to the sample tubes. Figure 4 shows sample tubes with reference stripes and screen.

Of the three coated tubes, one was covered with 100 mesh Stainless Steel 316 (SS316) screen, one was covered with 100 mesh Ni200 screen, and one was not covered with screen. Of the three uncoated tubes, one was covered with 100 mesh SS316 screen, one was covered with 100 mesh Ni200 screen, and one was not covered with screen. For the screen coated sample tubes, a single wrap of screen was tightly wrapped around the tube and spot welded together at the point of overlap. The screen was also spot welded to the coated or uncoated tube using the fewest number of welds sufficient to hold the screen in place. The screen was attached after the initial measurements of the tube diameter and removed before the final measurement of the tube diameter. Table 4 shows the sample tube test matrix.

After fabrication, the solubility corrosion test vehicles were charged with 680cc of high purity sodium 10-20 ppm oxygen). After charging, the test vehicles were processed at 1073K to remove non-condensable gases and then operated at 1073K for one hour. After processing, the test vehicles were then allowed to cool to 573K and the sodium charge was removed from the test vehicles through drain tubes into dump pots. After the sodium was removed, the test vehicles were charged again with 680cc of high purity sodium. After the second charge was loaded, the test vehicles were processed at 1073K to remove non-

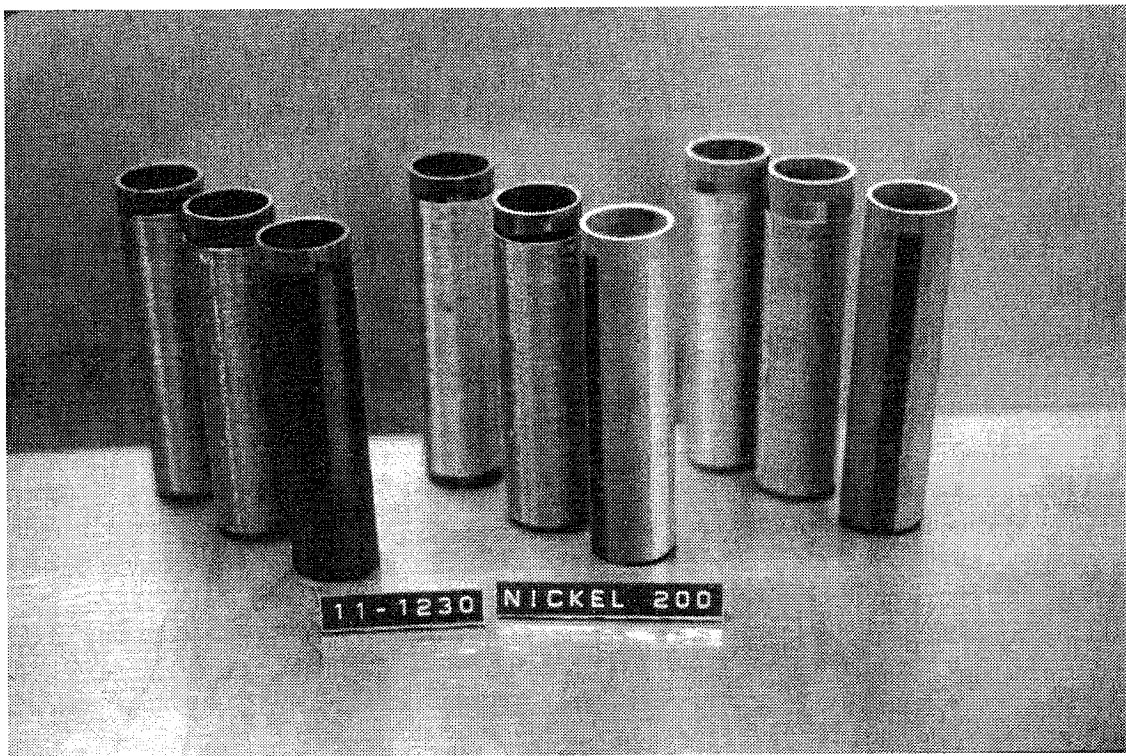


Figure 4. Uncoated and Nickel Aluminide Coated Ni200 Sample Tubes with Reference Stripe and Screen.

condensable gases and operated at 1073K for one hour. After the second processing step, the test vehicles were again allowed to cool to 573K and a small amount of sodium was allowed to fill the drain tubes. The drain tubes were pinched and welded at both ends. These samples (not analyzed) were retained for possible analyses later in the program. After the samples were removed, the test vehicles were operated at 1073K for a period of up to 8760 cumulative hours.

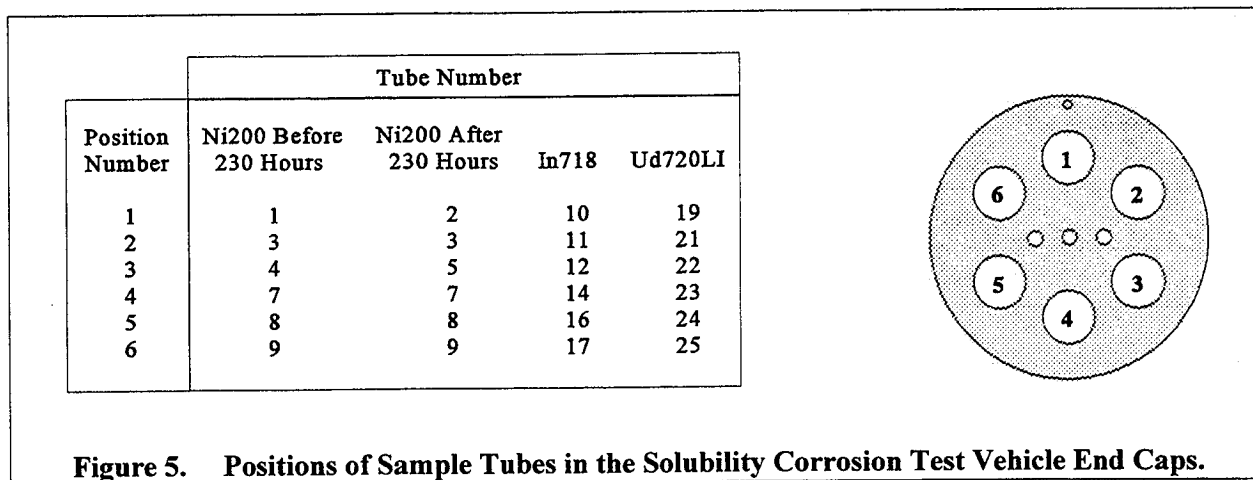
At the end of the testing, the sample tubes were removed from each test vehicle, and measured to determine the depth and rate of corrosion (if any) of the tube surfaces. After being measured, the tubes were sectioned, photomicrographed, and analyzed.

Nickel 200 Solubility Corrosion Test Vehicle

In June 1994, the Ni200 solubility corrosion test vehicle was charged with sodium and processed as discussed above. After processing, the test vehicle was operated at 1073K for approximately 65 hours. At 65 hours, a leak was detected in the Ni200 sample tube-to-Ni200 end cap weld joint at Position No. 3. Figure 5 shows the positions of the sample tubes in the end caps of the solubility corrosion test vehicles. The test vehicle was cooled and backfilled with argon to prevent oxidation of the sodium. The sample tube-to-end cap joint was ground and then rewelded using In61 filler material. After rewelding, the test vehicle was reprocessed to remove non-condensable gases, and operation of the test vehicle at 1073K continued.

Table 4. Sample Tube Test Matrix for Solubility Corrosion Tests.

Tube No.	Tube Material	Coating Material	Screen Material	Tube Use
1	Ni200	Nickel Aluminide	None	Tested 230 hours
2	Ni200	Nickel Aluminide	None	Tested 2270 hours
3	Ni200	Nickel Aluminide	SS316	Tested 2500 hours
4	Ni200	Nickel Aluminide	Ni200	Tested 230 hours
5	Ni200	Nickel Aluminide	Ni200	Tested 2270 hours
6	Ni200	None	None	Not Tested
7	Ni200	None	None	Tested 2500 hours
8	Ni200	None	SS316	Tested 2500 hours
9	Ni200	None	Ni200	Tested 2500 hours
10	In718	Nickel Aluminide	None	Tested 8767 hours
11	In718	Nickel Aluminide	SS316	Tested 8767 hours
12	In718	Nickel Aluminide	Ni200	Tested 8767 hours
13	In718	Nickel Aluminide	Ni200	Not Tested
14	In718	None	None	Tested 8767 hours
15	In718	None	None	Not Tested
16	In718	None	SS316	Tested 8767 hours
17	In718	None	Ni200	Tested 8767 hours
18	Ud720LI	Nickel Aluminide	SS316	Not Tested
19	Ud720LI	Nickel Aluminide	None	Tested 1886 hours
20	Ud720LI	Nickel Aluminide	Ni200	Not Tested
21	Ud720LI	Nickel Aluminide	SS316	Tested 1886 hours
22	Ud720LI	Nickel Aluminide	Ni200	Tested 1886 hours
23	Ud720LI	None	None	Tested 1886 hours
24	Ud720LI	None	SS316	Tested 1886 hours
25	Ud720LI	None	Ni200	Tested 1886 hours
26	Ud720LI	None	None	Not Tested



After the test vehicle was operated for approximately 230 hours, a leak was detected again in the Ni200 sample tube-to-Ni200 end cap weld joint at Position No. 3. The test vehicle was cooled and backfilled with argon, and the joint ground and rewelded using Inconel 61 filler material. After rewelding the test vehicle was not leak tight.

For assistance, Inco International, Inc. was contacted and recommended either In61 or In82 for welding Ni200 (sample tube) to Ni200 (end cap). Based on its lower aluminum and titanium contents and increased physical properties, In82 was selected over In61. Table 5 lists the aluminum and titanium contents, tensile and yield strengths, and elongations for In61 and In82. Using In82 filler material, the joint was again ground and rewelded. However, after rewelding, the test vehicle was still not leak tight.

After these several unsuccessful attempts to reweld the joint, Thermacore replaced the Ni200 end cap with a SS316 end cap, because Thermacore had more experience welding SS316 to Ni200 than welding Ni200 to Ni200. The sample tubes were removed from the Ni200 end cap and welded into the SS316 end cap. Sample Tube No. 4 was damaged during the rewelding attempts and replaced with Sample Tube No. 5. In addition, the reference stripes on Sample Tube No. 1 partially detached from the nickel aluminide coating during testing. As a result, Sample Tube No. 1 was replaced with Sample Tube No. 2. After the tubes were welded into the SS316 end cap, the end cap was welded into the Ni200 test vehicle.

Table 5. Chemical Compositions and Physical Properties for In61 and In82 Filler Materials.

	Inconel 61	Inconel 82
% Aluminum / % Titanium	1.5 max. / 2.0 to 3.5	N/A / 0.75
Tensile Strength, MPa (ksi)	413 (60)	552 (80)
0.2% Yield Strength, MPa (ksi)	241 (35)	276 (40)
Elongation, %	20	30

In three steps, the repaired test vehicle was charged with sodium and processed at 1073K to remove non-condensable gases. After each processing step, the sodium charge was removed, and clean sodium was pushed into the test vehicle. After the third processing step, the test vehicle was cooled to 573K and a sodium sample (not analyzed) was removed for possible analysis later in the program.

Based on data taken over the full period of testing, the Ni200 sample tubes were tested at an average temperature of 1077K with a standard deviation of 2K. The condensation heat flux incident upon the Ni200 tubes was 27.9W/cm² with a standard deviation of 3.2W/cm². At least once per week, data were recorded.

In November 1994, operation of the Ni200 test vehicle was stopped at 2500 hours because of a sodium leak in a Ni200 sample tube-to-SS316 end cap weld joint at Position No. 1. During the preliminary evaluation of the leak site, it was observed that the affected tube had broken loose and fallen into the test vehicle. At this point, the Ni200 test vehicle was covered with a plastic layer and purged with argon. Thermacore and NASA LeRC discussed disposal and/or repair options for this test vehicle and decided that the test vehicle should not be repaired.

At this time, the sample tubes were removed from the end cap and measured. The measurements for the Ni200 sample tubes before and after testing are included in Appendix B. The measurements indicated that these sample tubes became distorted (oval-shaped) during testing and, thus, these measurements could not be used to determine corrosion rates. The Ni200 sample tubes, the SS316 and Ni200 screens, and the weld joint local to the leak area were sectioned, photomicrographed and analyzed. Table 6 shows the results for the screens. Several typical photomicrographs for the screen samples are included in Appendix C. The photomicrographs showing the edge of the reference stripe were used to determine the rate of corrosion and several typical examples are included in Appendix D. Table 7 shows the results for the stripe edge samples. Table 8 shows the results for the center of reference stripe and coating samples. Several photomicrographs for these samples are included in Appendix E. The sample number identifies the photomicrograph samples. In the tables, the first number in the sample number refers to the task number (e.g., "2" refers to Task 2). Sample locations are referenced from the test vehicle end cap (see Figure 1).

The photomicrographs for the screen samples showed visible signs of corrosion for the layer of Ni200 screen wrapped on Sample Tubes Nos. 5 and 9 and showed no visible signs of corrosion for the layer of SS316 screen wrapped on Sample Tubes Nos. 3 and 8. There was no attempt to measure the depth of corrosion on the Ni200 screens due to the lack of a suitable reference.

The photomicrographs for all sample tubes were taken at distances of 1.0, 2.0 and 3.0 inches from the end cap (Figure A3, Appendix A) and at several locations around the circumference (a,b,c,d and e) of the tubes at these distances. Note that the post-test coating thicknesses shown in Tables 7 and 8 vary from about 0.001 to 0.008 cm. As described previously, the initial coating thickness was 0.008 cm, but this was then reduced in the final centerless grinding operation. Finally, some of the photomicrographs, such as for samples 79 and 94a of Appendix E, show an extra layer on top of the coating or reference stripe; this is a result of a contaminant in the potting compound used to prepare the sample for photomicrography.

The difficulty in determining the depth of corrosion from the photomicrographs should also be noted. In many instances, the reference stripe disbonded partially or totally from the sample tubes. Some of this happened during testing, but most of the disbonding occurred during sample preparation for the photomicrographs. A judgment was made for each edge photomicrograph as to whether enough of the stripe remained to form a good measurement reference. There were also some difficulties in determining the exact

Table 6. Analyses of SS316 and Ni200 Screens from Ni200, In718 and Ud720LI Solubility Corrosion Test Vehicles.

Sample Numbers	Tube Material and Number	Sample Description	Hours Tested	Wire Diameter before Test, cm	Corrosion
2-25, 2-26, 2-27	Nickel 3	SS316 screen, 1.0in, 2.0in & 3.0in from end cap	2500	0.010	no visible signs
2-28, 2-29, 2-30	Nickel 5	Ni200 screen, 1.0in, 2.0in & 3.0in from end cap	2270	0.010	visible signs; no reference from which to measure
2-31, 2-32, 2-33	Nickel 8	SS316 screen, 1.0in, 2.0in & 3.0in from end cap	2500	0.010	no visible signs
2-34, 2-35, 2-36	Nickel 9	Ni200 screen, 1.0in, 2.0in & 3.0in from end cap	2500	0.010	visible signs; no reference from which to measure
2-97, 2-98, 2-99	Inconel 11	SS316 screen, 1.0in, 2.0in & 3.0in from end cap	8767	0.010	no visible signs
---	Inconel 12	Ni200 screen	8767	0.010	screen reduced to dust
2-100, 2-101, 2-102	Inconel 16	SS316 screen, 1.0in, 2.0in & 3.0in from end cap	8767	0.010	no visible signs
---	Inconel 17	Ni200 screen	8767	0.010	screen reduced to dust
2-37, 2-38, 2-39	Udimet 21	SS316 screen, 1.0in, 2.0in & 3.0in from end cap	1886	0.010	no visible signs
2-40, 2-41a,b, 2-42a,b	Udimet 22	Ni200 screen, 1.0in, 2.0in & 3.0in from end cap	1886	0.010	visible signs; no reference from which to measure
2-43	Udimet 24	SS316 screen, 1.0in from end cap	1886	0.010	visible signs; no reference from which to measure
2-44, 2-45		SS316 screen, 2.0in & 3.0in from end cap			no visible signs
2-46, 2-47, 2-48	Udimet 25	Ni200 screen, 1.0in, 2.0in & 3.0in from end cap	1886	0.010	visible signs; no reference from which to measure

Table 7. Analyses of Edge of Reference Stripe Samples from Ni200, In718 and Ud720LI Solubility Corrosion Test Vehicles (After Testing).

Sample No.	Tube Material / No.	Sample Description	Hours	Coating Thick., cm	Stripe Thick., cm	Corrosion
2-1a edge	Nickel 1	NiAl coated, 4 W stripes, 1.0in from end cap	230	0.00318	detached from coating	no reference line
2-2a edge		NiAl coated, 4 W stripes, 2.0in from end cap		0.00318	detached from coating	no reference line
2-3a edge		NiAl coated, 4 W stripes, 3.0in from end cap		0.00318	detached from coating	no reference line
2-3b edge				0.00396	detached from coating	no reference line
2-4a1 edge	Nickel 2	NiAl coated, 4 W stripes, 1.0in from end cap	2270	0.00079	0.00079 partially detached	no visible signs
2-4b1 edge				0.00318	0.00635	0.0061 cm/yr
2-5a edge		NiAl coated, 4 W stripes, 2.0in from end cap		0.00318	0.00635	0.0061 cm/yr
2-6 edge		NiAl coated, 4 W stripes, 3.0in from end cap		0.00318	0.00635 partially detached	no reference line
2-7a1 edge	Nickel 3	NiAl coated, 4 W stripes, 1.0in from end cap	2500	0.00635	detached from coating	no reference line
2-7b1 edge				0.00792	detached from coating	no reference line
2-8 edge		NiAl coated, 4 W stripes, 2.0in from end cap		0.00635	detached from coating	no reference line
2-9b edge		NiAl coated, 4 W stripes, 3.0in from end cap		0.00318	0.00318	0.0112 cm/yr
2-10a edge	Nickel 5	NiAl coated, 4 W stripes, 1.0in from end cap	2270	0.00318	detached from coating	no reference line
2-11b edge		NiAl coated, 4 W stripes, 2.0in from end cap		0.00318	detached from coating	no reference line
2-12a edge		NiAl coated, 4 W stripes, 3.0in from end cap		0.00318	0.00318	0.0030 cm/yr
2-12b1 edge				0.00475	0.00318	no visible signs
2-16a edge	Nickel 7	Uncoated 2W/2Ni stripes, 1.0in from end cap	2500	N/A	0.00157	0.0056 cm/yr
2-16b edge					trace of stripe remaining	no reference line
2-16c edge					0.00157	no visible signs
2-17c edge		Uncoated 2W/2Ni stripes, 2.0in from end cap			0.00157 - 0.00318 partially detached	no reference line
2-17d edge					0.00157	0.0056 cm/yr
2-18a edge		Uncoated 2W/2Ni stripes, 3.0in from end cap			detached from substrate	no reference line
2-18b edge					0.00157	0.0028 cm/yr

Table 7. Analyses of Edge of Reference Stripe Samples from Ni200, In718 and Ud720LI Solubility Corrosion Test Vehicles (After Testing, Continued).

Sample No.	Tube Material / No.	Description	Hours	Coating Thick., cm	Stripe Thick, cm.	Corrosion
2-21a edge	Nickel 8	Uncoated 2W/2Ni stripes, 3.0in from end cap	2500	N/A	0.00157 - 0.00318	0.0056 cm/yr
2-21b edge					0.00318	0.0028 cm/yr
2-22a edge	Nickel 9	Uncoated 2W/2Ni stripes, 1.0in from end cap	2500	N/A	0.00239	0.0028 cm/yr
2-22b edge					0.00157	0.0028 cm/yr
2-23a edge		Uncoated 2W/2Ni stripes, 2.0in from end cap			0.00157	0.0028 cm/yr
2-23b edge					0.00157	0.0056 cm/yr
2-24a edge		Uncoated 2W/2Ni stripes, 3.0in from end cap			0.00318	0.0028 cm/yr
2-24b edge					0.00318	no visible signs
2-73b edge	Inconel 10	NiAl coated, 4 W stripes, 1.0in from end cap	8767	0.00157	0.00157	no visible signs
2-74b edge		NiAl coated, 4 W stripes, 2.0in from end cap		0.00157	0.00079	0.00079 cm/yr
2-75b edge		NiAl coated, 4 W stripes, 3.0in from end cap		0.00157	0.00079	no visible signs
2-76b edge	Inconel 11	NiAl coated, 4 W stripes, 1.0in from end cap	8767	0.00157	0.00079	no visible signs
2-77b edge		NiAl coated, 4 W stripes, 2.0in from end cap		0.00157	0.00079 - 0.00157	no visible signs
2-78b edge		NiAl coated, 4 W stripes, 3.0in from end cap		0.00079	0.00079	no visible signs
2-85b edge	Inconel 14	Uncoated, 4 W stripes, 1.0in from end cap	8767	N/A	0.00157	no visible signs
2-87b edge		Uncoated, 4 W stripes, 3.0in from end cap			0.01270 split	no visible signs
2-94b edge	Inconel 17	Uncoated, 4 W stripes, 1.0in from end cap	8767	N/A	0.01270	no visible signs
2-95b edge		Uncoated, 4 W stripes, 2.0in from end cap			0.01016	no visible signs
2-96b edge		Uncoated, 4 W stripes, 3.0in from end cap			0.01016	no visible signs
2-52 edge	Udimet 19	NiAl coated, 4 W stripes, 1.0in from end cap	1886	0.00079 - 0.00157	detached from coating	no reference line
2-53 edge		NiAl coated, 4 W stripes, 2.0in from end cap		0.00079 - 0.00157	detached from coating	no reference line
2-54 edge		NiAl coated, 4 W stripes, 3.0in from end cap		0.00079	detached from coating	no reference line

Table 7. Analyses of Edge of Reference Stripe Samples from Ni200, In718 and Ud720LI Solubility Corrosion Test Vehicles (After Testing, Continued).

Sample No.	Tube Material / No.	Description	Hours	Coating Thick., cm	Stripe Thick., cm	Corrosion
2-55 edge	Udimet 21	NiAl coated, 4 W stripes, 1.0in from end cap	1886	0.00157	detached from coating	no reference line
2-56 edge		NiAl coated, 4 W stripes, 2.0in from end cap		0.00157	split within thickness	no visible signs
2-57 edge		NiAl coated, 4 W stripes, 3.0in from end cap		0.00079	split within thickness	no visible signs
2-58 edge	Udimet 22	NiAl coated, 4 W stripes, 1.0in from end cap	1886	0.00157 - 0.00239	split within thickness	no visible signs
2-59 edge		NiAl coated, 4 W stripes, 2.0in from end cap		0.00157 - 0.00239	0.00318 split	no visible signs
2-60 edge		NiAl coated, 4 W stripes, 3.0in from end cap		0.00079 - 0.00239	0.00157 - 0.00318	no visible signs
2-62 edge	Udimet 23	Uncoated, 4 W stripes, 2.0in from end cap	1886	N/A	split within thickness	no visible signs
2-63 edge		Uncoated, 4 W stripes, 3.0in from end cap			split within thickness	no reference line
2-64 edge	Udimet 24	Uncoated, 4 W stripes, 1.0in from end cap	1886	N/A	split within thickness	no visible signs
2-65 edge		Uncoated, 4 W stripes, 2.0in from end cap			detached from substrate	no reference line
2-66 edge		Uncoated, 4 W stripes, 3.0in from end cap			detached from substrate	no reference line
2-67 edge	Udimet 25	Uncoated, 4 W stripes, 1.0in from end cap	1886	N/A	0.00157 - 0.01270	no visible signs
2-68 edge		Uncoated, 4 W stripes, 2.0in from end cap			0.01422	no visible signs
2-69 edge		Uncoated, 4 W stripes, 3.0in from end cap			0.00157 - 0.01110	no visible signs

Table 8. Analyses of Center of Reference Stripe and Coating Samples from Ni200, In718 and Ud720LI Solubility Corrosion Test Vehicles (After Testing).

Sample No.	Tube Material / No.	Description	Hours	Coating Thick., cm	Stripe Thick., cm
2-1b coating	Nickel 1	NiAl coated, 4 W stripes, 1.0in from end cap	230	0.00396	not shown
2-2b coating		NiAl coated, 4 W stripes, 2.0in from end cap		0.00318	not shown
2-3c coating		NiAl coated, 4 W stripes, 3.0in from end cap		0.00396	not shown
2-3d coating				0.00318	not shown
2-4a2 coating	Nickel 2	NiAl coated, 4 W stripes, 1.0in from end cap	2270	0.00318	not shown
2-4b2 coating		NiAl coated, 4 W stripes, 1.0in from end cap		0.00318	not shown
2-5b center		NiAl coated, 4 W stripes, 2.0in from end cap		0.00318	0.00318
2-7a2 coating	Nickel 3	NiAl coated, 4 W stripes, 1.0in from end cap	2500	0.00635	not shown
2-7b2 coating		NiAl coated, 4 W stripes, 1.0in from end cap		0.00635	not shown
2-9a coating		NiAl coated, 4 W stripes, 3.0in from end cap		0.00475	not shown
2-10b coating	Nickel 5	NiAl coated, 4 W stripes, 1.0in from end cap	2270	0.00318 - 0.00475	not shown
2-11a coating		NiAl coated, 4 W stripes, 2.0in from end cap		0.00635	not shown
2-12b2 coating		NiAl coated, 4 W stripes, 3.0in from end cap		0.00635	not shown
2-17a center	Nickel 7	Uncoated 2W/2Ni stripes, 2.0in from end cap	2500	N/A	0.00475 - 0.00635
2-17b center					0.00318 - 0.00475
2-19a center	Nickel 8	Uncoated 2W/2Ni stripes, 1.0in from end cap	2500	N/A	0.00318
2-19b center					trace - 0.00318
2-20a center	Nickel 8	Uncoated 2W/2Ni stripes, 2.0in from end cap	2500	N/A	trace - 0.00318
2-20b center					trace
2-73a center	Inconel 10	NiAl coated, 4 W stripes, 1.0in from end cap	8767	0.00239	0.00157
2-74a center		NiAl coated, 4 W stripes, 2.0in from end cap		0.00239	0.00157
2-75a center		NiAl coated, 4 W stripes, 3.0in from end cap		0.00157	0.00079

Table 8. Analyses of Center of Reference Stripe and Coating Samples from Ni200, In718 and Ud720LI Solubility Corrosion Test Vehicles (After Testing, Continued).

Sample No.	Tube Material / No.	Description	Hours	Coating Thick., cm	Stripe Thick., cm
2-76a center	Inconel 11	NiAl coated, 4 W stripes, 1.0in from end cap	8767	0.00157	0.00157
2-77a center		NiAl coated, 4 W stripes, 2.0in from end cap		0.00157	0.00157
2-78a center		NiAl coated, 4 W stripes, 3.0in from end cap		0.00079	0.00079
2-79 center	Inconel 12	NiAl coated, 4 W stripes, 1.0in from end cap	8767	0.00157	0.01270
2-80 center		NiAl coated, 4 W stripes, 2.0in from end cap		0.00239	0.00239 - 0.00318
2-81 center		NiAl coated, 4 W stripes, 3.0in from end cap		0.00318	0.00318
2-85a center	Inconel 14	Uncoated, 4 W stripes, 1.0in from end cap	8767	N/A	0.00157
2-86 center		Uncoated, 4 W stripes, 2.0in from end cap			0.00079
2-87a center		Uncoated, 4 W stripes, 3.0in from end cap			0.01270 split
2-91 center	Inconel 16	Uncoated, 4 W stripes, 1.0in from end cap	8767	N/A	0.00157
2-92 center		Uncoated, 4 W stripes, 2.0in from end cap			0.00157
2-93 center		Uncoated, 4 W stripes, 3.0in from end cap			0.00157
2-94a center	Inconel 17	Uncoated, 4 W stripes, 1.0in from end cap	8767	N/A	0.01270
2-95a center		Uncoated, 4 W stripes, 2.0in from end cap			0.01016
2-96a center		Uncoated, 4 W stripes, 3.0in from end cap			0.01016
2-61 center	Udimet 23	Uncoated, 4 W stripes, 1.0in from end cap	1886	N/A	split within thickness

locations on the photomicrographs of the reference stripe and coating surfaces and in judging the effect of tube curvature in photomicrographs that were magnified only 100 times (compared to those done at 200x magnification). In some cases, the disbonded reference stripe appeared to pull away part of the nickel aluminide coating from the tube. These problems were encountered in varying degrees for samples from each of the solubility corrosion test vehicles.

As determined from the photomicrographs, the results for the Ni200 sample tubes are as follows: (see Table 4 for a list of coatings and screen materials):

- For Sample Tube No. 1 (tested 230 hours, replaced with Sample Tube No.2):
 - At 1.0in, 2.0in and 3.0in (a, b) from the end cap, the reference stripe detached from the nickel aluminide coating; no reference line remained.
- For Sample Tube No. 2 (tested 2270 hours):
 - At 1.0in (a), no visible signs of corrosion.
 - At 1.0in (b), visible signs of corrosion. Estimated corrosion was 0.0061cm/yr.
 - At 2.0in, visible signs of corrosion. Estimated corrosion was 0.0061cm/yr.
 - At 3.0in, the reference stripe partially detached from the nickel aluminide coating; no reference line remained.
- For Sample Tube No. 3 (tested 2500 hours):
 - At 1.0in (a, b) and 2.0in, the reference stripe detached from the nickel aluminide coating; no reference line remained.
 - At 3.0in, visible signs of corrosion. Estimated corrosion was 0.0112cm/yr.
- Sample Tube No. 4 (tested 230 hours, replaced with Sample Tube No. 5) was damaged during the rewelding attempts and was not analyzed.
- For Sample Tube No. 5 (tested 2270 hours):
 - At 1.0in and 2.0in, the reference stripe detached from the nickel aluminide coating; no reference line remained.
 - At 3.0in (a), visible signs of corrosion. Estimated corrosion was 0.0030cm/yr.
 - At 3.0in (b), no visible signs of corrosion.
- Sample Tube No. 6 was not tested.
- For Sample Tube No. 7 (tested 2500 hours):
 - At 1.0in (a), visible signs of corrosion. Estimated corrosion was 0.0056cm/yr.
 - At 1.0in (b), trace of reference stripe remained attached to the Ni200 substrate; no reference line remained.
 - At 1.0in (c), no visible signs of corrosion.
 - At 2.0in (a), the reference stripe partially detached from the Ni200 substrate; no

reference line remained.

- At 2.0in (b), visible signs of corrosion. Estimated corrosion was 0.0056cm/yr.
- At 3.0in (a), the reference stripe detached from the Ni200 substrate; no reference line remained.
- At 3.0in (b), visible signs of corrosion. Estimated corrosion was 0.0028cm/yr.

■ For Sample Tube No. 8 (tested 2500 hours):

- At 1.0in, no sample.
- At 2.0in, no sample.
- At 3.0in (a), visible signs of corrosion. Estimated corrosion was 0.0056cm/yr.
- At 3.0in (b), visible signs of corrosion. Estimated corrosion was 0.0028cm/yr.

■ For Sample Tube No. 9 (tested 2500 hours):

- At 1.0in (a), visible signs of corrosion. Estimated corrosion was 0.0028cm/yr.
- At 1.0in (b), visible signs of corrosion. Estimated corrosion was 0.0028cm/yr.
- At 2.0in (a), visible signs of corrosion. Estimated corrosion was 0.0056cm/yr.
- At 2.0in (b), visible signs of corrosion. Estimated corrosion was 0.0056cm/yr.
- At 3.0in (a), visible signs of corrosion. Estimated corrosion was 0.0028cm/yr.
- At 3.0in (b), no visible signs of corrosion.

In summary, for the Ni200 sample tubes after approximately 2500 hours of testing in sodium at 1073K, the photomicrographs showed no visible signs of corrosion for the uncoated SS316 screen and moderate corrosion for the uncoated Ni200 screen. Based on twelve edge of reference stripe samples, the uncoated Ni200 sample tubes showed an average corrosion rate of 0.0033cm/yr with a standard deviation of 0.0019cm/yr. Only two of the twelve samples showed no corrosion. The results for the nickel aluminide coated Ni200 sample tubes were based on only six edge of reference stripe samples and were much less consistent than the uncoated sample tube results. The average corrosion rate for the six samples was 0.0044cm/yr with a standard deviation of 0.0039cm/yr. The possible cause of the corrosion of the leak area weld joint was sodium attack of the oxides within the weld joint which formed during TIG welding of the test vehicle.

Inconel 718 Solubility Corrosion Test Vehicle

In July 1994, the In718 test vehicle was charged with sodium and processed as discussed previously. During processing, leaks were detected in the In718 sample tube-to-In718 end cap weld joints at Positions Nos. 2 and 3. Figure 5 shows the positions of the sample tubes in the end caps of the solubility corrosion test vehicles. The test vehicle was cooled and backfilled with argon to prevent oxidation of the sodium. The sample tube-to-end cap joints were ground and then rewelded using In718 filler material. After rewelding, the test vehicle was reprocessed to remove non-condensable gases, and operation of the test vehicle at 1073K began.

After the test vehicle was operated for approximately 6600 hours, a pinhole leak was detected at the In718 sample tube-to-In718 end cap weld joint at Position No. 2. The leak was repaired and operation of the test vehicle continued.

Based on data taken over the full period of testing, the In718 sample tubes were tested at an average temperature of 1076K with a standard deviation of 3K. The condensation heat flux incident upon the In718 tubes was 26.7W/cm² with a standard deviation of 2.9W/cm². At least once per week, data were recorded.

In August 1995, operation of the In718 test vehicle was stopped at 8767 hours (8760 goal). At this time, the sample tubes were removed from the end cap and measured. The measurements for the In718 sample tubes before and after testing are included in Appendix B. The measurements indicated that these sample tubes also became distorted during testing and, thus, these measurements could not be used to determine corrosion rates. The In718 sample tubes and SS316 and Ni200 screens were sectioned, photomicrographed and analyzed. Table 6 includes the results for the screens. Several typical photomicrographs for the screen samples are included in Appendix C. The photomicrographs showing the edge of the reference stripe were used to determine presence of corrosion and several typical examples are included in Appendix D. Table 7 shows the results for the stripe edge samples. Table 8 shows the results for the center of reference stripe and coating samples. Several photomicrographs for these samples are included in Appendix E.

The Ni200 screen was reduced to dust during testing, indicating extensive corrosion of the layer of Ni200 screen wrapped on Sample Tubes Nos. 12 and 17. The photomicrographs showed no visible signs of corrosion for the layer of SS316 screen wrapped on Sample Tubes Nos. 11 and 16.

As determined from the photomicrographs, the results for the In718 sample tubes are as follows (see Table 4 for a list of coating and screen material on each tube):

- For Sample Tube No.10 (tested 8767 hours):
 - At 1.0in, no visible signs of corrosion.
 - At 2.0in, visible signs of corrosion. Estimated corrosion was 0.0079cm/yr.
 - At 3.0in, no visible signs of corrosion.
- For Sample Tube No. 11 (tested 8767 hours):
 - At 1.0in, 2.0in and 3.0in, no visible signs of corrosion.
- For Sample Tube No. 12 (tested 8767 hours):
 - At 1.0in, 2.0in and 3.0in, no photomicrographs showing edge of reference stripe. Not possible to determine corrosion.
- Sample Tube No. 13 was not tested.
- For Sample Tube No. 14 (tested 8767 hours):
 - At 1.0in, no visible signs of corrosion.
 - At 2.0in, no photomicrographs showing edge of reference stripe. Not possible to determine corrosion.
 - At 3.0in, no visible signs of corrosion.

- Sample Tube No. 15 was not tested.
- For Sample Tube No. 16 (tested 8767 hours):
 - At 1.0in, 2.0in and 3.0in, no photomicrographs showing edge of reference stripe. Not possible to determine corrosion.
- For Sample Tube No. 17 (tested 8767 hours):
 - At 1.0in, 2.0in and 3.0in, no visible signs of corrosion.

In summary, for the In718 sample tubes after 8767 hours of testing in sodium at 1073K, the photomicrographs showed no visible signs of corrosion for the uncoated SS316 screen and severe deterioration of the uncoated Ni200 screen. Out of six edge of reference stripe samples there was one possible indication of corrosion for the nickel aluminide coated In718 sample tubes; the others showed no visible signs of corrosion. Based on five edge of reference stripe samples, there were no visible signs of corrosion for the uncoated In718 sample tubes.

Udimet 720LI Solubility Corrosion Test Vehicle

In July 1994, six Ud720LI sample tubes were brazed into an In718 end cap. Figure 5 shows the positions of the sample tubes in the end cap. Initially, the assembly was vacuum brazed at 1315K for fifteen minutes using Nicrobraz LM as the filler material. The braze schedule is discussed in Section 2.2.3. After brazing, the braze joints were not helium leak tight and an oxide layer had formed on the end cap and sample tubes. As a result, the assembly was rebrazed at 1315K for fifteen minutes using a small amount of additional Nicrobraz LM filler material. After rebrazing, the assembly was helium leak tight, but some of the oxide layer remained. This remaining oxide may have increased the rate of corrosion during testing. However, the post-test results indicated essentially no corrosion on these sample tubes.

During brazing and rebrazing of the sample tube/end cap assembly, a layer of oxidized tungsten disbonded from three reference stripes on the sample tubes at Positions Nos. 2 and 6 and from four reference stripes on the sample tube at Position No. 3. The oxidized layer remained bonded to the reference stripes on the sample tubes at Positions Nos. 1, 4 and 5. The thickness of the disbonded layers was approximately one-half to two-thirds of the 0.013cm thickness of the original plasma-sprayed stripe. As a result, the tungsten layers which remained bonded to the sample tubes at Positions Nos. 2, 3 and 6 had thicknesses of approximately 0.005cm. After discussing the situation, Thermacore and NASA LeRC decided that the reduced tungsten layers should have sufficient thicknesses for use as reference stripes. Therefore, fabrication of the Ud720LI test vehicle continued.

After fabrication, the test vehicle was charge with sodium and processed as discussed above. After processing, the test vehicle was operated at 1073K for 1610 hours. At 1610 hours, a leak was detected in the Ud720LI sample tube-to-In718 end cap braze joint at Position No. 3. Thermacore and NASA LeRC decided to weld a cap over the braze joint leak area including the entire sample tube. Therefore, this sample tube was no longer subjected to the high heat flux condenser load. After the cap was welded over the joint area, the test vehicle was reprocessed. The reprocessing consisted of removing the old sodium fluid charge; filling with a cleaning charge; operating at 1073K for one hour; removing the cleaning charge; filling with

a second fluid charge; and processing the test vehicle to remove non-condensable gases. After reprocessing, operation of the test vehicle continued.

Based on data taken over the full period of testing, the Ud720LI sample tubes were tested at an average temperature of 1074K with a standard deviation of 3K. The condensation heat flux incident upon the Ud720LI tubes was 28.7W/cm² with a standard deviation of 5.6W/cm². At least once per week, data were recorded.

In January 1995, operation of the Ud720LI test vehicle was stopped at 1886 hours because of a sodium leak in the In718 end cap-to-SS316 wall weld joint and a second leak in a Ud720LI sample tube-to-In718 end cap braze joint. Thermacore and NASA LeRC discussed repair options and decided that the test vehicle should not be repaired. At this time, the sample tubes were removed from the end cap and measured. The measurements for the Ud720LI sample tubes before and after testing are included in Appendix B. The measurements indicated that these sample tubes also became distorted during testing. The Ud720LI sample tubes and SS316 and Ni200 screens were sectioned, photomicrographed and analyzed. Table 6 includes the results for the screens. Several typical photomicrographs for the screen samples are included in Appendix C. The photomicrographs showing the edge of the reference stripe were used to determine presence of corrosion and several typical examples are included in Appendix D. Table 7 shows the results for the stripe edge samples. Table 8 shows the results for the center of reference stripe and coating samples.

The photomicrographs for the screen samples showed visible signs of corrosion for the layer of Ni200 screen wrapped on Sample Tubes Nos. 22 and 25, and only one of six samples showed visible signs of corrosion for the layer of SS316 screen wrapped on Sample Tubes Nos. 21 and 24.

As determined from the photomicrographs, the results for the Ud720LI sample tubes are as follows (see Table 4 for a list of coating and screen material on each tube):

- Sample Tube No. 18 was not tested.
- For Sample Tube No. 19 (tested 1886 hours):
 - At 1.0in, 2.0in and 3.0in, the reference stripe detached from the nickel aluminide coating; no reference line remained.
- Sample Tube No. 20 was not tested.
- For Sample Tube No. 21 (tested 1886 hours):
 - At 1.0in, stripe detached from the nickel aluminide coating; no reference line remained.
 - At 2.0in and 3.0in, no visible signs of corrosion.
- For Sample Tube No. 22 (tested 1886 hours):
 - At 1.0in, 2.0in and 3.0in, no visible signs of corrosion.
- For Sample Tube No. 23 (tested 1886 hours):

- At 1.0in, no photomicrographs showing edge of reference stripe.
 - At 2.0in, no visible signs of corrosion.
 - At 3.0in, stripe detached from the Ud720LI substrate; no reference line remained.
- For Sample Tube No. 24 (tested 1886 hours):
 - At 1.0in, no visible signs of corrosion.
 - At 2.0in and 3.0in, the reference stripe detached from the Ud720LI substrate; no reference line remained.
 - For Sample Tube No. 25 (tested 1886 hours):
 - At 1.0in, 2.0in and 3.0in, no visible signs of corrosion.
 - Sample Tube No. 26 was not tested.

In summary for the Ud720LI sample tubes after approximately 1886 hours of testing in sodium at 1073K, one of the six photomicrographs showed visible signs of corrosion for the uncoated SS316 screen while the other five showed no visible corrosion. The photomicrographs showed moderate corrosion for the uncoated Ni200 screen. Based on five edge of reference stripe samples for each, there were no visible signs of corrosion for the nickel aluminide coated Ud720LI sample tubes or for the uncoated Ud720LI sample tubes. The possible cause of the corrosion of the leak area weld joint was sodium attack of the oxides within the weld joint which formed during TIG welding of the test vehicle. The possible cause of the corrosion of the leak area braze joint was sodium attack of the oxides within the braze joint which formed during initial brazing of the end cap.

2.2.2 Reference Stripe Materials

As discussed in Section 2.2.1, a test vehicle was designed to provide sodium vapor for condensation on test samples at a 25 W/cm² heat flux. To determine the corrosion on a surface, a part of the surface must be protected as a reference from which differences can be measured. This section discusses the reference stripe selection process which was based on Phase I experience and the evaluation of diffusion of the reference stripe material candidates into the substrate material and thermal expansion differences between the stripe and substrate materials.

In Phase I, each Ni200 sample tube was prepared with four equally spaced electroless plated nickel reference stripes. After testing, the reference lines between the stripes and the uncoated sample tubes remained intact. However, the nickel stripes diffused into the nickel aluminide on the coated sample tubes. As a result, alternative stripe materials were required, at least, for the coated Phase II sample tubes.

In Phase II, each sample tube was prepared with four equally spaced reference stripes to provide reference lines from which to scale photomicrographs. The reference stripes were 0.013cm thick, 0.64cm wide and 9.53cm long. On the uncoated and nickel aluminide coated In718 and Ud720LI sample tubes, the four reference stripes were plasma-sprayed tungsten. On the nickel aluminide coated Ni200 sample tubes, the four reference stripes were also plasma-sprayed tungsten. On the uncoated Ni200 sample tubes, two reference stripes were plasma-sprayed tungsten and two were electroless plated nickel. The plasma-sprayed

tungsten was selected based on the results of the diffusion and thermal cycling tests discussed in this section. The electroless plated nickel was selected based on its successful Phase I use and because it would not significantly diffuse into the Ni200 tube. Table 3 shows the reference stripe materials that were applied to the sample tubes. This section details the tests performed prior to selecting the reference stripe materials.

Aluminum oxide was the first material investigated as it is believed to be compatible with sodium and would not diffuse into nickel aluminide. Hitemco deposited plasma-sprayed aluminum oxide reference stripes onto two nickel aluminide coated and two uncoated In718 substrate specimens. The reference stripes were 0.64cm wide, 5.1cm long and 0.013cm thick. Visual inspection of the four specimens indicated that the aluminum oxide stripes were bonded to the coated and uncoated substrates. Two specimens, one coated and one uncoated, were vacuum fired at 1073K for eight hours. After firing, the furnace was cooled to 313K. The specimens were cycled five times between 1073K and 313K. After five cycles, the aluminum oxide stripe was still attached to the uncoated substrate but not attached to the coated substrate. All four specimens were sectioned, photomicrographed, and analyzed. Photomicrographs for the nickel aluminide coated and uncoated specimens are included in Appendix F. The photomicrographs indicated that the aluminum oxide stripe cracked and delaminated from the nickel aluminide coated In 718 substrate specimen after vacuum firing. The photomicrographs also indicated that the aluminum oxide stripe remained bonded to the uncoated In718 substrate specimen after vacuum firing.

Hitemco stated that the delamination of the aluminum oxide stripe was most likely a result of not being sufficiently bonded to the smooth surface of the nickel aluminide coating along with a thermal expansion mismatch between the two materials. In an attempt to improve bonding, Hitemco prepared another nickel aluminide coated In718 substrate specimen with a 0.64cm wide and 5.1cm long stripe. The plasma-sprayed aluminum oxide was deposited onto a grit-blasted area of the coated substrate. This specimen was thermally cycled from 313K to 1073K and again delaminated from the coating. As a result, plasma-sprayed aluminum oxide was judged to be unfeasible for use as a reference stripe material.

In January 1994, alternative materials were evaluated for use as reference stripe materials. Several candidate materials are shown in Table 9. The primary evaluation parameters included sodium corrosion resistance, the rate of diffusion in aluminum, the rate of diffusion in nickel, the coefficient of thermal expansion, and the melting point. Derived from Fick's Second Law, Equation (1), can be used to determine the concentration of one solid material within another as a function of depth and time^[15].

$$\frac{C_s - C_x}{C_s - C_0} = \operatorname{erf} \left(\frac{x}{2\sqrt{Dt}} \right) \quad (1)$$

where

C_s \equiv concentration at the surface,
 C_0 \equiv initial concentration in the material,
 C_x \equiv concentration at a depth x ,
 x \equiv depth (cm),
 t \equiv time (seconds),
 D \equiv diffusion coefficient (cm^2/s).

Table 9. Sodium Corrosion Resistance of Reference Stripe Materials at 1073K^[16-25].

Material	Sodium Corrosion Resistance	Material	Sodium Corrosion Resistance
Zirconium	Good	Tungsten	Good
Tantalum	Good	Chromium	Good
Hafnium	Good	Osmium	Limited
Niobium	Good	Vanadium	Good
Ruthenium	Good	Cobalt	Good
Molybdenum	Good	Iron	Good
Rhenium	Good	Nickel	Good
Titanium	Limited	Platinum	Poor

First, the materials were evaluated based on their resistance to sodium corrosion at 1073K. Table 9 shows the materials and the associated sodium corrosion resistances. In the table, "good" indicates a corrosion rate less than 0.003cm/yr; "limited" indicates a corrosion rate greater than 0.003cm/yr but less than 0.025cm/yr; and "poor" indicates a corrosion rate greater than 0.025cm/yr. Titanium, osmium and platinum with corrosion rates greater than 0.003cm/yr were eliminated from further evaluation.

Each of the remaining materials was then evaluated based on its coefficient of thermal expansion (CTE) and melting point with respect to the CTE and melting point of In718. The goal was to select candidate materials with CTEs and melting points similar to In718. The coefficients of thermal expansion, the CTE ratios, the melting points, and the melting point ratios are included in Appendix G. Chromium was selected, because data for diffusion in nickel aluminide was available from NASA LeRC. In Phase I nickel was shown to diffuse readily into nickel aluminide, and, as a result, was eliminated for use on coated sample tubes. Cobalt and iron are similar to nickel and were also eliminated. Availability and cost of the powder required to plasma spray the reference stripes onto the sample tubes narrowed the choices to tungsten, rhenium and molybdenum with vanadium as an alternative. These materials were also expected to have low rates of diffusion into the sample tubes and the nickel aluminide coating. Table 10 shows the reference stripe material test matrix.

Thermal Cycling Tests

Eight uncoated and eight nickel aluminide coated In718 specimens were fabricated. Two uncoated and two coated specimen were dedicated to a single candidate reference stripe material. One plasma-sprayed stripe was deposited on each specimen. Two specimens, one uncoated and one coated specimen for each material, were thermal cycled in vacuum five times between 293K and 1073K. The specimens were placed into the furnace and heated to 1073K for at least eighteen hours. The furnace was then cooled to 293K and the specimens were removed and photographed. Figure 6 shows plasma-sprayed reference stripes on uncoated and nickel aluminide coated In718 substrates. Table 11 shows the visual inspection results for the thermal cycling tests. The results of the cycle tests are discussed below.

Table 10. Candidate Reference Stripe Material Test Matrix.

Sample Number	Stripe Material	In718 Substrate Coating	Thermal Cycle Tested	Thermal Diffusion Tested
1	Tungsten	NiAl	Yes	No
2			No	Yes
3	Rhenium	NiAl	Yes	No
4			No	Yes
5	Molybdenum	NiAl	Yes	No
6			No	Yes
7	Chromium	NiAl	Yes	No
8			No	Yes
9	Tungsten	None	Yes	No
10			No	Yes
11	Rhenium	None	Yes	No
12			No	Yes
13	Molybdenum	None	Yes	No
14			No	Yes
15	Chromium	None	Yes	No
16			No	Yes

Table 11. Thermal Cycling Test Results for Candidate Reference Stripe Materials.

Sample Number	Stripe Material	In718 Substrate Coating	Condition of Stripe after Each 1073K Cycle				
			1	2	3	4	5
1	Tungsten	NiAl	Bonded	Bonded	Bonded	Bonded	Bonded
2		None	Bonded	Bonded	Bonded	Bonded	Bonded
3	Rhenium	NiAl	Bonded	Bonded	Bonded	Bonded	Bonded
4		None	Bonded	Bonded	Bonded	Bonded	Bonded
5	Molybdenum	NiAl	Bonded	Bonded	Bonded	Bonded	3/4 bonded
6		None	Bonded	Bonded	Bonded	Bonded	Bonded
7	Chromium	NiAl	Disbonded after one cycle; end of test				
8		None	Bonded	Bonded	Bonded	Bonded	Bonded

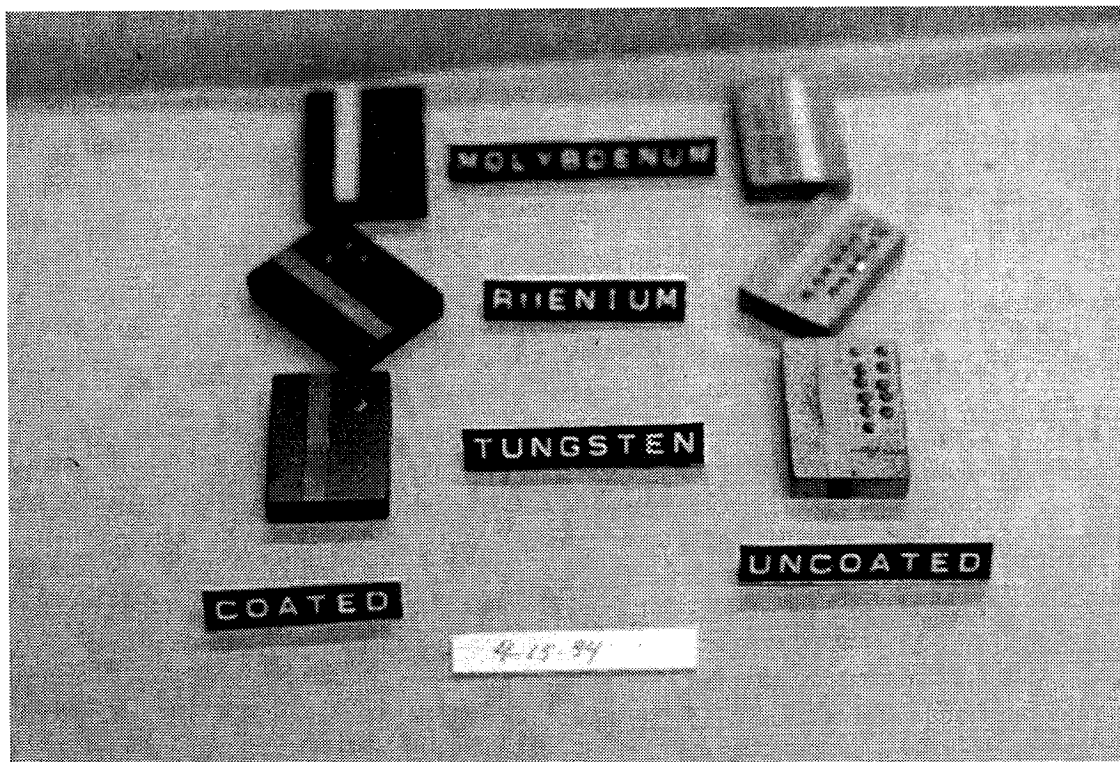


Figure 6. Plasma-Sprayed Reference Stripes on Uncoated and Nickel Aluminide Coated In718 Substrates.

After one cycle between 293K and 1073K, the chromium stripe was disbonded from the nickel aluminide coated In718 substrate. After five cycles between 293K and 1073K, the chromium stripe remained bonded to the uncoated In718 substrate.

After five cycles between 293K and 1073K, the tungsten stripes remained bonded to the nickel aluminide coated and uncoated In718 substrates.

After four cycles between 293K and 1073K, the molybdenum stripe remained bonded to the nickel aluminide coated and uncoated In718 substrates. After five cycles between 293K and 1073K, three-quarters of the molybdenum stripe was disbonded from the nickel aluminide coated In718 substrate. However, the molybdenum stripe remained bonded to the uncoated In718 substrate.

After five cycles between 293K and 1073K, the rhenium stripes remained bonded to the nickel aluminide coated and uncoated In718 substrates.

Thermal Diffusion Tests

Since data were available for the diffusion of chromium into nickel aluminide, chromium was selected as a baseline to which the thermal diffusion tests could be compared. Evaluation of the data showed that chromium should diffuse into nickel aluminide to a depth of 0.014cm at a temperature of 1073K after

8760 hours; to a depth of 0.022cm at a temperature of 1273K after 300 hours; and to a depth of 0.12cm at a temperature of 1273K after 8760 hours. Thus, accelerated diffusion tests could be run at 1273K for about 200 hours to simulate an 8760 hour test at 1073K. The calculations for these predictions are included in Appendix H.

Two specimens, one uncoated and one coated specimen for each material, were vacuum fired at 1073K for 200 hours. After 200 hours, the specimens were cut into two pieces. The first piece was sectioned, photomicrographed and analyzed. The second piece was vacuum fired at 1273K for an additional 200 hours. The photomicrographs were used to determine the diffusion rate of each material in nickel aluminide and In718. Tables 12 and 13 show the visual inspection and photomicrograph results for the 200 hour diffusion tests. Photomicrographs for the 200 hour, chromium and tungsten diffusion tests are included in Appendix I. The results of the 200 hour diffusion tests are discussed below.

After 400 hours, the diffusion test specimens were sectioned, photomicrographed and analyzed. Tables 12 and 14 show the visual inspection and photomicrograph results for the 400 hour diffusion tests. Photomicrographs for the 400 hour, chromium and tungsten diffusion tests are also included in Appendix I. The results of the 400 hour diffusion tests are discussed below.

At 143 hours, the chromium stripes remained bonded to the nickel aluminide coated and uncoated In718 substrates. The specimens were returned to the furnace for 64 hours of additional testing. At 207 hours, the chromium stripes remained bonded to the nickel aluminide coated and uncoated In718 substrates. Each chromium sample was split into two pieces. Two halves, one nickel aluminide coated and one uncoated, were analyzed. Photomicrographs for the 200 hour diffusion test indicated that essentially no diffusion of the chromium into the nickel aluminide coating or the uncoated In718 had occurred. The remaining halves were returned to the furnace for 200 hours of additional testing.

The photomicrographs of the 200 hour chromium diffusion samples showed no diffusion of chromium into nickel aluminide. Two possible explanations for this unexpected result are:

- During plasma spraying of the chromium stripe, a chromium oxide layer formed on the particles and inhibited the diffusion of chromium into nickel aluminide.
- The contact area between the chromium particles of the reference stripe and the nickel aluminide coating was small. As a result, the amount of chromium diffusion over time was significantly reduced.

To verify either explanation, additional analyses of the 200 hour chromium diffusion samples were required. However, the purpose of these tests was to evaluate candidates for use as reference stripe materials. The materials were required to have a good sodium corrosion resistance; a CTE which nearly matches that of nickel aluminide or the tube material; a good bond between the stripe and the nickel aluminide or the tube material; and a low rate of diffusion into nickel aluminide or the tube material. The thermal cycling and diffusion tests accomplished this task and no further analyses of the chromium samples were required.

At 343 hours, the chromium stripes remained bonded to the nickel aluminide coated and uncoated In718 substrates. The substrate halves were returned to the furnace for 57 hours of additional testing. At 405 hours, the chromium stripes remained bonded to the nickel aluminide coated and uncoated In718 substrate. The chromium stripe disbonded from the uncoated In718 substrate half while the sample was

Table 12. Visual Inspection Results for Diffusion Tests of Reference Stripe Materials.

Sample Number	Stripe Material	In718 Substrate Coating	Condition of Stripe after Diffusion at 1273K			
			143 Hours	207 Hours	343 Hours	405 Hours
1	Chromium	NiAl	Bonded	Bonded	Bonded	Bonded
2	Chromium	None	Bonded	Bonded	Bonded	Bonded
			64 Hours	200 Hours	261 Hours	421 Hours
3	Tungsten	NiAl	Bonded	Bonded	Bonded	Bonded
4	Tungsten	None	Bonded	Bonded	Bonded	Bonded
5	Molybdenum	NiAl	Disbonded after 64 hours; end of test			
6	Molybdenum	None	Bonded	Bonded	Bonded	Disbonded
7	Rhenium	NiAl	Disbonded after 64 hours; end of test			
8	Rhenium	None	Bonded	3/4 Disbonded	Bonded	Disbonded

Table 13. Photomicrograph Results for 200 Hour Diffusion Test of Reference Stripe Materials.

Sample Number	Stripe Material and Thickness (cm)	In718 Substrate Coating and Thickness (cm)	Diffusion Test Conditions	Diffusion into Coating or Substrate (cm)
1	Chromium, 0.0127	NiAl, 0.0097	1273K, 207 hr	None
2	Chromium, 0.0097	None	1273K, 207 hr	None
3	Tungsten, 0.0127	NiAl, 0.0097	1273K, 200 hr	None
4	Tungsten, 0.0112	None	1273K, 200 hr	0.0008
5	Molybdenum, 0.0140	NiAl, 0.0064	1273K, 64 hr	None
6	Molybdenum, 0.0127	None	1273K, 200 hr	0.0008
7	Rhenium, 0.0127	NiAl, 0.0079	1273K, 64 hr	None
8	Rhenium, 0.0127	None	1273K, 200 hr	None

Table 14. Photomicrograph Results for 400 Hour Diffusion Test of Reference Stripe Materials.

Sample Number	Stripe Material and Thickness (cm)	In718 Substrate Coating and Thickness (cm)	Diffusion Test Conditions	Diffusion into Coating or Substrate (cm)
1	Chromium, 0.0102	NiAl, 0.0152	1273K, 405 hr	None
2	Chromium, disbonded	None	1273K, 405 hr	0.0025
3	Tungsten, 0.0102	NiAl, 0.0102	1273K, 421 hr	None
4	Tungsten, 0.0114	None	1273K, 421 hr	0.0013
5	Molybdenum, 0.0140	NiAl, 0.0064	1273K, 64 hr	None
6	Molybdenum; disbonded	None	1273K, 421 hr	0.0013
7	Rhenium, 0.0127	NiAl, 0.0079	1273K, 64 hr	None
8	Rhenium; disbonded	None	1273K, 421 hr	0.0008

prepared for photomicrography. Photomicrographs for the 400 hour diffusion tests indicated that essentially no diffusion of the chromium into the nickel aluminide coating had occurred. Photomicrographs also indicated that chromium had diffused into the uncoated In718 substrate to a depth of approximately 0.0025cm.

At 64 hours, the tungsten stripes remained bonded to the nickel aluminide coated and uncoated In718 substrates. The samples were returned to the furnace for 136 hours of additional testing. At 200 hours, the tungsten stripes remained bonded to the nickel aluminide coated and uncoated In718 substrates. Each tungsten sample was split into two pieces. Two halves, one nickel aluminide coated and one uncoated, were analyzed. Photomicrographs for the 200 hour diffusion test indicated that essentially no diffusion of the tungsten into the nickel aluminide coating had occurred. Photomicrographs also indicated that tungsten diffused into the uncoated In718 substrate to a depth of 0.0008cm. The remaining halves were returned to the furnace for 200 hours of additional testing.

At 261 hours, the tungsten stripes remained bonded to the nickel aluminide coated and uncoated In718 substrates. The substrate halves were returned to the furnace for 139 hours of additional testing. At 421 hours, the tungsten stripes remained bonded to the nickel aluminide coated and uncoated In718 substrates. Photomicrographs for the 400 hour diffusion tests indicated that essentially no diffusion of the tungsten into the nickel aluminide coating had occurred. Photomicrographs also indicated that tungsten diffused into the uncoated In718 substrate to a depth of 0.0013cm.

At 64 hours, the molybdenum stripe was disbonded from the nickel aluminide coated In718 substrate. This sample was split into two pieces. One half was analyzed to determine the depth of molybdenum diffusion into the nickel aluminide coating. Photomicrographs for the 64 hour diffusion test indicated that essentially no diffusion of the molybdenum into nickel aluminide coating had occurred. The molybdenum stripe remained bonded to the uncoated In718 substrate. The uncoated sample was returned to the furnace for 136 hours of additional testing. At 200 hours, the molybdenum stripe remained bonded to the uncoated

In718 substrate. The molybdenum sample was split into two pieces. One half was analyzed. Photomicrographs indicated that molybdenum diffused into the uncoated In718 substrate to a depth of 0.0008cm. The remaining half was returned to the furnace for 200 hours of additional testing.

At 261 hours, the molybdenum stripe remained bonded to the uncoated In718 substrate. The substrate was returned to the furnace for 139 hours of additional testing. At 421 hours, the molybdenum stripe was disbonded from the uncoated In718 substrate. Photomicrographs for the 400 hour diffusion test indicated that molybdenum diffused into the uncoated In718 substrate to a depth of 0.0013cm.

At 64 hours, the rhenium stripe was disbonded from the nickel aluminide coated In718 substrate. This sample was split into two pieces. One half was analyzed. Photomicrographs for the 64 hour diffusion test indicated that essentially no diffusion of the rhenium into the nickel aluminide coating had occurred. The rhenium stripe remained bonded to the uncoated In718 substrate. The uncoated sample was returned to the furnace for 136 hours of additional testing. At 200 hours, the rhenium stripe was 3/4 disbonded from the uncoated In718 substrate. The rhenium sample was split into two pieces - the 3/4 section on which the stripe was disbonded and the remaining 1/4 section with the stripe still bonded. The 3/4 piece was analyzed. Photomicrographs for the 200 hour diffusion test indicated that essentially no diffusion of the rhenium into the uncoated In718 had occurred. The remaining quarter was returned to the furnace for 200 hours of additional testing.

At 261 hours, the rhenium stripe remained bonded to the uncoated In718 substrate. The substrate quarter was returned to the furnace for 139 hours of additional testing. At 421 hours, the rhenium stripe was disbonded from the uncoated In718 substrate quarter. Photomicrographs for the 400 hour diffusion test indicated that rhenium diffused into the uncoated In718 substrate to a depth of 0.0008cm.

Based on the thermal cycling and diffusion test results, Thermacore and NASA LeRC decided to apply four plasma-sprayed tungsten reference stripes to the nickel aluminide coated Ni200, In718 and Ud720LI sample tubes and to the uncoated In718 and Ud720LI sample tubes. Two plasma-sprayed tungsten reference stripes and two electroless plated nickel reference stripes were selected for the uncoated Ni200 sample tubes.

During the Task 2 tests of the solubility corrosion test vehicles, the electroless plated nickel reference stripes on the uncoated Ni200 sample tubes were used successfully to maintain a reference line from which to measure corrosion. The tungsten reference stripes were used with mixed results. The stripes provided a good reference when they remained attached to the sample tubes. However, on many of the sample tubes, the reference stripes disbonded from the substrate during testing or preparation of the sample tube sections for analyses. Possible solutions to minimize these difficulties include using a thinner reference stripe (less than 0.013cm) to minimize thermal stresses, modifying sample tube preparation to reduce the damage to the stripes, and/or selecting an alternative stripe material.

2.2.3 Udimet 720/ Inconel 718 Brazing and Welding Experiments

Before joining six Ud720LI sample tubes to an In718 sample tube sheet for Solubility Corrosion Test Vehicle No. 3, several brazing and welding experiments were performed to demonstrate the ability to join Ud720LI and In718. High strength superalloys such as Ud720 can not generally be joined by conventional fusion welding techniques due to cracking problems. Table 15 shows the matrix of joining experiments. Figures 7 and 8 show braze and weld test articles, respectively.

Table 15. Test Matrix for the Udimet 720LI / Inconel 718 Brazing and Welding Experiments.

Sample Number	Method of Joining	Procedure
1 and 2	Furnace braze	Electrolytic nickel plated Udimet 720LI. (0.0013cm thick.) Unplated Inconel 718. Filler material: Microbraz LM Brazing temperature: 1313K ; Duration: 15 Minutes
3 and 4	Furnace braze	Electrolytic nickel plated Udimet 720LI. (0.0013cm thick.) Electrolytic nickel plated Inconel 718. (0.0013cm thick.) Filler material: Microbraz LM Brazing temperature: 1313K ; Duration: 15 Minutes
1 and 2	Electron beam weld	Unplated Udimet 720LI. Unplated Inconel 718. Electron beam welder: Advanced Technology
3 and 4	Electron beam weld	Unplated Udimet 720LI. Unplated Inconel 718. Electron beam welder: Applied Energy

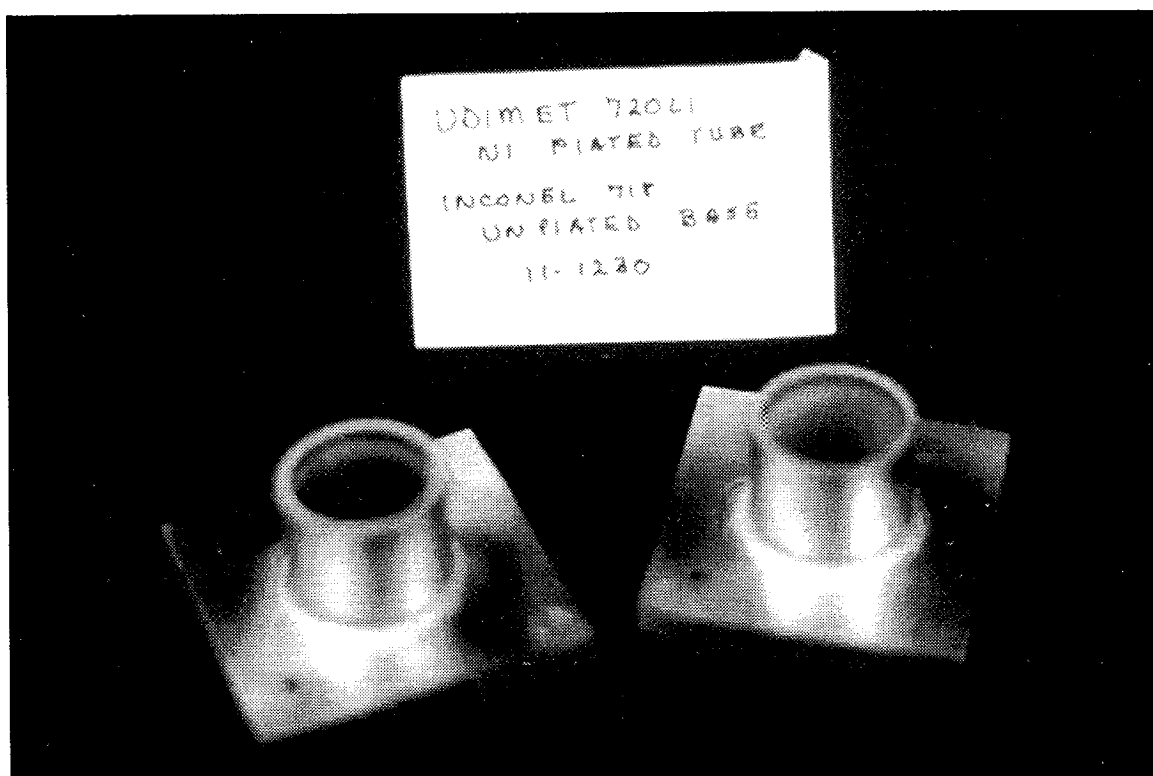


Figure 7. Nickel Plated Ud720LI Tube to Unplated In718 Plate Braze Samples (Nos. 1 & 2).

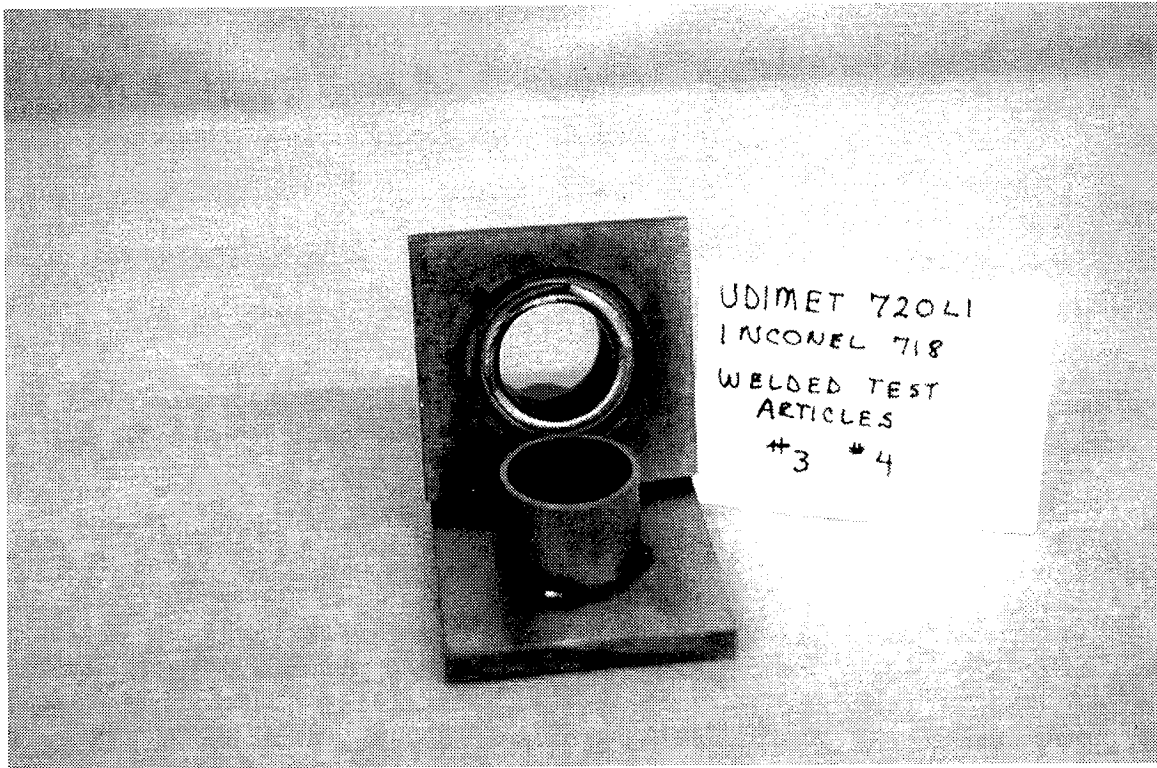


Figure 8. Unplated Ud720LI Tube to Unplated In718 Plate Weld Samples (Nos. 3 & 4).

Prior to the start of the joining tests, Thermacore discussed the proposed joining techniques with cognizant personnel in the industry, such as application engineers at Special Metals Corporation, materials specialists at NASA LeRC, and with Mechanical Technology, Inc. (MTI) fabrication engineers who have worked with these materials.

Preliminary investigations indicated that Ud700 had been brazed to René 80 at approximately 1473K. The parts were held at temperature for 25 minutes. The filler material was BNi-1 (AWS Specification) which is similar to Nicrobraz 125. Additional investigations indicated that Udimet 720 could be joined to In718 and Ud720LI using a process called Transient Liquid Phase Diffusion Bonding (TLPDB). TLPDB is a joining process which uses a liquid interface to achieve bonding of the material surfaces^[26-28]. In addition, MTI had attempted electron-beam welding Ud720LI to In718, Ud720LI to Ud720LI, and Ud720LI to other materials. MTI had not successfully welded fine grain or coarse grain Ud720LI to In718 nor Ud720LI to Ud720LI. However, MTI has successfully welded fine grain (but not coarse grain) Ud720LI to In617^[29].

In November 1993, the Ud720LI/In718 brazing and welding experiments were completed. Results of the sample brazing and welding experiments are discussed below and summarized in Table 16.

The first braze experiment consisted of joining an electrolytic nickel plated Ud720LI tube to an unplated Inconel 718 plate. Two assemblies (1 and 2) were brazed using the sample braze procedure in Appendix J. The filler material was Nicrobraz LM (BNi-2). The assemblies were brazed at 1313K for fifteen minutes. After the furnace run, the two assemblies were helium leak checked and were leak tight.

Table 16. Results for the Udimet 720/Inconel 718 Brazing and Welding Experiments.

Method of Joining	Sample Number	Helium Leak Tight	Vacuum Fired	Helium Leak Tight
Furnace brazing	1	Yes	No	N/A
	2	Yes	1373K, 4 hours	Yes
	3	Yes	No	N/A
	4	Yes	1373K, 4 hours	Yes
Electron beam welding	1	No	No	N/A
	2	No	1373K, 4 hours	No
	3	No	No	N/A
	4	No	1373K, 4 hours	No

Assembly 2 was then vacuum fired at 1373K for four hours. After firing, Assembly 2 was again helium leak checked and was still leak tight. Assemblies 1 and 2 were sectioned, photomicrographed, and analyzed. Photomicrographs for this braze procedure are included in Appendix J. The photomicrographs indicated that the Microbrazed LM filler material wetted to both the unplated In718 and to the plated Ud720LI.

The second braze experiment consisted of joining an electrolytic nickel plated Ud720LI tube to an electrolytic nickel plated In718 plate. Two assemblies (3 and 4) were brazed using the sample braze procedure in Appendix J. The filler material was Microbrazed LM. The assemblies were brazed at 1313K for fifteen minutes. After the furnace run, the two assemblies were helium leak checked. Assembly 3 was leak tight and Assembly 4 was not leak tight. Assembly 4 with added Microbrazed LM filler material was brazed a second time at 1323K. The assembly was held at temperature for fifteen minutes. After the second furnace run, Assembly 4 was helium leak checked and was leak tight. Assembly 4 was vacuum fired at 1373K for four hours. After firing, Assembly 4 was again helium leak checked and was still leak tight. Assemblies 3 and 4 were sectioned, photomicrographed, and analyzed. Photomicrographs for this braze procedure are also included in Appendix J. The photomicrographs indicate that the Microbrazed LM filler material wetted to both the plated In718 and to the plated Ud720LI.

The first weld experiment consisted of joining an unplated Ud720LI tube to an unplated In718 plate. Two assemblies (1 and 2) were electron beam welded at Advanced Technology (Pasadena CA) using the sample weld procedure in Appendix K. After welding, the two assemblies were helium leak checked and were not leak tight. Assembly 2 was vacuum fired at 1373K for four hours. After firing, assembly 2 was again helium leak checked to determine if the size of the leak had changed. The assembly was not leak tight and the size of the leak did not change. Assemblies 1 and 2 were sectioned, photomicrographed and analyzed. Photomicrographs for this weld procedure are included in Appendix K. The photomicrographs indicate that a full penetration weld was not achieved.

The second weld experiment also consisted of joining an unplated Ud720LI tube to an unplated In718 plate. Two assemblies (3 and 4) were electron beam welded at Applied Energy (Winchester MA) using the sample weld procedure in Appendix K. After welding, the two assemblies were helium leak

Table 17. Chemical Compositions for Selected Nickel-based Superalloys.

Composition	Udimet 720	Ud 720LI	Udimet 700	Inconel 718	Inconel 617	René 80
Ni	Balance	Balance	Balance	52.5	Balance	Balance
Cr	18.0	16.0	15.5	19.0	22.0	14.0
Co	14.7	14.7	17.0	0.5	12.5	9.5
Mo	3.0	3.0	5.5	3.1	9.0	4.0
Al	2.5	2.5	4.0	0.5	1.0	3.0
Ti	5.0	5.0	3.7	0.9	0.6	5.0
C	0.035	0.010	0.05	0.04	0.07	0.17
B	0.033	0.015	0.025	0.003	0.006	0.02
W	1.25	1.25	--	--	--	4.0
Zr / Fe	0.03	0.03	0.03	--	--	0.06
Fe	0.08	0.08	2.0	Balance	3.0	0.18

checked and were not leak tight. Assembly 4 was vacuum fired at 1373K for four hours. After firing, Assembly 4 was again helium leak checked to determine if the size of the leak had changed. The assembly was not leak tight and the size of the leak did not change. Assemblies 3 and 4 were sectioned, photomicrographed and analyzed. Photomicrographs for this weld procedure are also included in Appendix K. The photomicrographs indicate that a full penetration weld was not achieved.

As a result of the brazing and welding experiments, Thermacore decided to braze plated Ud720LI sample tubes into the plated In718 sample end cap.

2.2.4 Availability of Udimet 720

After contacting approximately twenty companies, AAA Metals Company, Inc., Hanson, MA was identified as a source for limited quantities of Ud720. Ud720 is available in at least two grades. The first grade, Ud720LI (Low Interstitial), has a fine grain structure and has the nominal chemical composition shown in Table 17. The second grade, Ud720CR (Creep Resistant), has a coarse grain structure and has a nominal chemical composition similar to Udimet 720LI. For both grades, the grain structure can be changed by heat treating the material. The nominal chemical compositions for Ud720^[30], Ud700^[26], In718^[31], In617^[32], and René 80^[33] are also shown in Table 17. Both Ud720LI and Ud720CR are readily available in mill runs of 1000 pounds or greater. However, small quantities of Ud720LI or Ud720CR are not readily stocked. The availability of small quantities of these materials is dependent upon the number of sample billets and/or the amount of excess material from a previous mill run.

In July 1993, Thermacore purchased 36in of 1.85in diameter Ud720LI round bar stock with a fine grain structure from AAA Metals Company, Inc. This material was used for evaluation and brazing and welding experiments (Section 2.2.3). In September, Thermacore contacted AAA Metals Company, Inc. to purchase additional Ud720LI round bar stock. At the time, AAA Metals had no Ud720LI in stock and could not locate any source for small quantities of Ud720LI. However, AAA Metals located several 14-15in sample billets of 2.0in diameter Ud720CR round bar stock with a coarse grain structure. Thermacore consulted with NASA LeRC and MTI. Based on these discussions and the results of the brazing and welding experiments, all parties agreed that Ud720LI with a fine grain structure was preferable for this application. In December 1993, AAA Metals Company located 18in of 5.0in diameter Ud720LI round bar stock with a fine grain structure. This material was purchased and used for the Ud720LI sample tubes.

2.3 TASK 3: APPLICATION OF COATINGS TO HEAT PIPE WICK STRUCTURES

The objective of Task 3 was to study the application of nickel aluminide coatings to heat pipe wick structures, namely capillary grooves and screens. In Phase I, the outer surfaces of cylindrical sample tubes were coated. In Phase II, coatings for capillary grooves and screens were also developed. The goal of this task was to develop and demonstrate complete coating of groove internals and screen wires. The technical approach for Task 3 is discussed in Sections 2.3.1 and 2.3.2.

The groove structures consisted of In718 parallel groove specimens with grooves of differing aspect ratios. Two methods were used to apply nickel aluminide coatings to the groove specimens. A two-step nickel aluminide application process produced coatings which were uniform and repeatable. Test pipes using sodium doped with aluminum powder produced coatings which were not uniform. Thermacore recommends using the two-step nickel aluminide application process for coating groove wick structures. The two coating methods evaluated for parallel groove wick structures are discussed in Section 2.3.1.

Since SS316 contained very little nickel for nickel aluminide formation, the screen specimens used two layers of 100 mesh or 60 mesh Ni200 screens. Three methods were evaluated to apply nickel aluminide coating to the screen specimens. A two-step aluminum-to-nickel aluminide application process produced coatings which were uniform, repeatable and relatively ductile. The two-step nickel aluminide application process produced coatings which were uniform and repeatable, but brittle. The one-step nickel aluminide application process produced coatings which were uniform and relatively brittle (more ductile than the two-step nickel aluminide coating), but less repeatable. Test pipes using sodium doped with aluminum powder produced coatings which were not uniform. Thermacore recommends using the two-step aluminum-to-nickel aluminide application process for coating screen wick structures and especially for those applications that require the more ductile screen for installation purposes. The one-step nickel aluminide application process should also be acceptable for heat pipes with simpler screen installation and where further high temperature processing of the heat pipe materials could lead to grain size and material property concerns. The three coating methods for screen wick structures are discussed in Section 2.3.2.

2.3.1 Parallel Groove Wick Structures

The following sections detail nickel aluminide application processes evaluated for parallel groove wick structures.

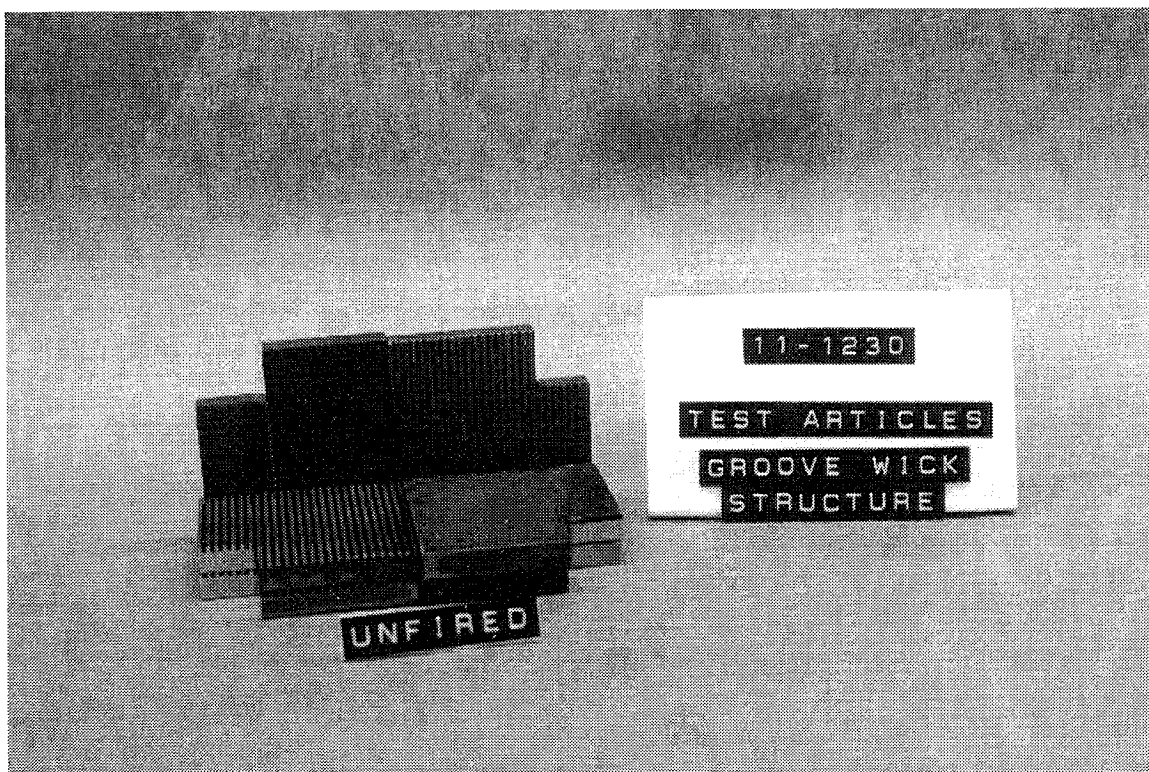


Figure 9. In718 Parallel Groove Specimens with Hitemco's Two-Step Nickel Aluminide Coating.

Two-step Nickel Aluminide Application Process

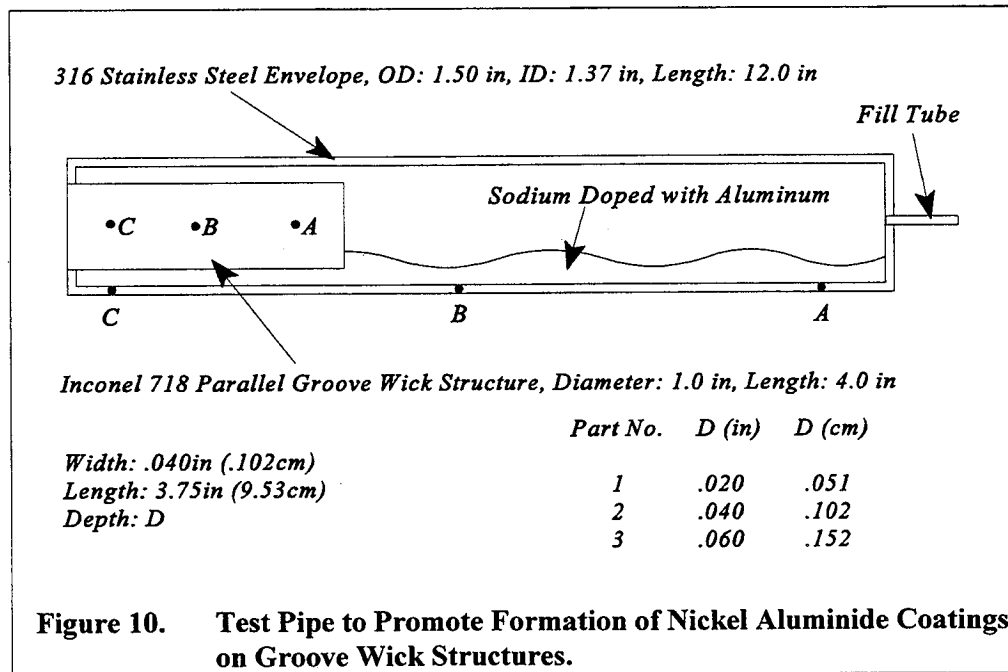
Two In718 specimens were machined for each of six parallel groove aspect ratios. Figure 9 shows a parallel groove specimen. After machining, Hitemco coated the In718 specimens using their two-step nickel aluminide application process. The first step was a four hour, 1193K pack cementation process. During this step, an essentially pure layer of aluminum was applied to the specimen. The second step was a diffusion heat treatment at 1313K. A coating thickness of approximately 0.013cm was applied to the groove specimens. After coating, one set of specimens was vacuum fired at 1073K for eight hours. In addition, one of the remaining specimens (sample no. 3) was vacuum fired at 1073K for twenty-four hours. After firing, all specimens were sectioned, photomicrographed and analyzed. Table 18 shows the test matrix and results for this coating method. Photomicrographs for samples 1, 3 and 6 are included in Appendix L. The photomicrographs indicated that approximately 0.013cm of nickel aluminide was formed on the parallel groove specimens, especially at the bottom of the groove structure where the minimum coating would be expected. The results show that even large aspect ratio grooves can be successfully coated.

Aluminum Doped Sodium Test Pipes for Coating Groove Specimens^[34-36]

As an alternative to forming the nickel aluminide coating on groove wick structures with the two-step nickel aluminide process, this method attempted to form nickel aluminide coating on In718 parallel groove wick structures within six closed SS316 test pipes containing an aluminum-doped sodium working fluid. Each Groove Test Pipe had one parallel groove specimen with 0.102cm wide and 0.051cm, 0.102cm or

Table 18. Test Matrix and Results for Parallel Groove Wick Structures Coated Using the Two-Step Nickel Aluminide Application Procedure.

Sample Number	Substrate Material	Groove Width, cm	Groove Depth, cm	Aspect Ratio d / w	Coating Thickness at Groove Bottom, cm	Fired at 1073K
1	In718	0.102	0.051	0.50	0.013	No
					0.015	Yes 8 hr
2	In718	0.102	0.076	0.75	0.013	No
					0.013	Yes 8 hr
3	In718	0.102	0.102	1.00	0.015	Yes 24 hr
					0.013	Yes 8 hr
4	In718	0.102	0.127	1.25	0.020	No
					0.013	Yes 8 hr
5	In718	0.102	0.152	1.50	0.013	No
					0.013	Yes 8 hr
6	In718	0.102	0.178	1.75	0.013	No
					0.013	Yes 8 hr



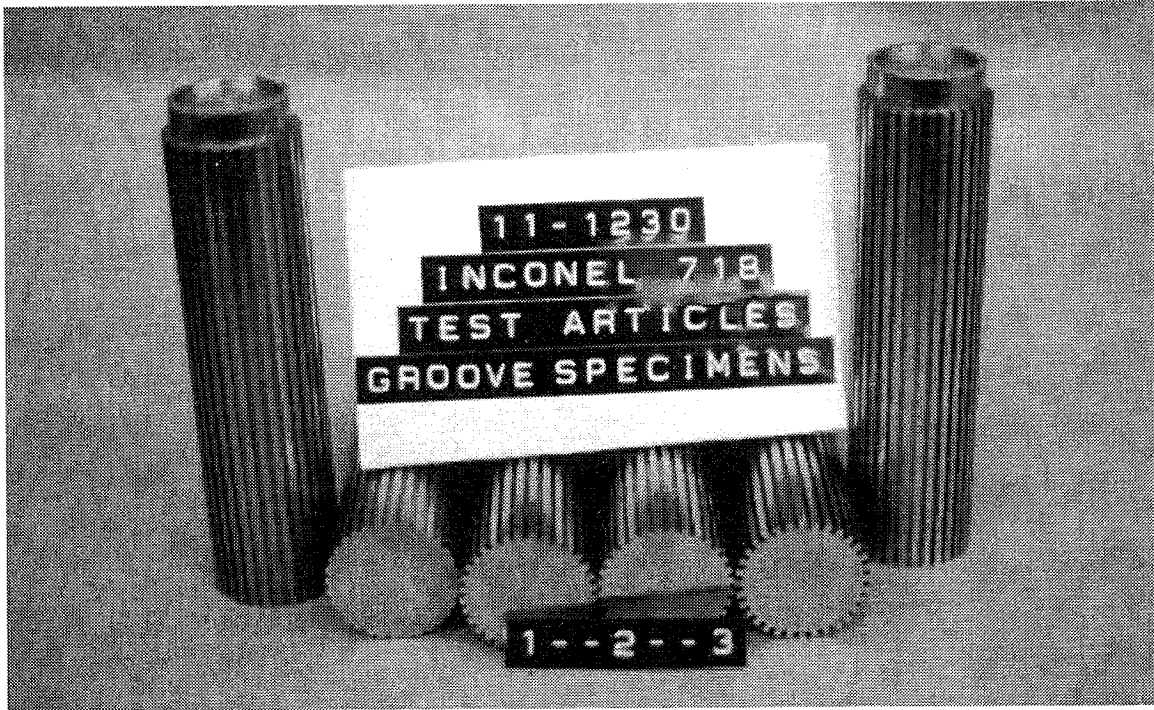


Figure 11. Uncoated In718 Parallel Groove Specimens for the Test Pipes with Aluminum Doped Sodium Working Fluid.

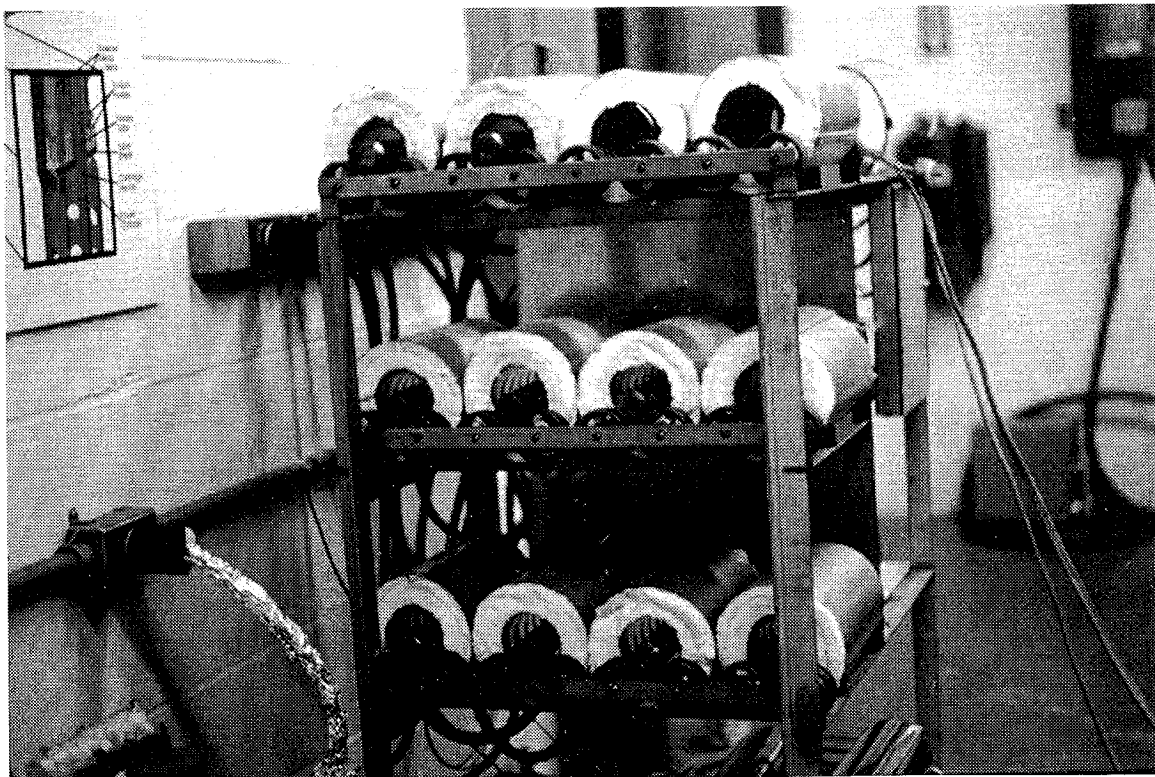


Figure 12. Groove and Screen Test Pipes Operating at 1073K and One Revolution Per Minute.

Table 19. Test Matrix for Coating Parallel Groove Wick Structures in Test Pipes Using Sodium Doped with Aluminum Powder.

Test Pipe No.	Groove Specimen		Test Pipe Operating Conditions	Amount of Sodium, g and Aluminum, g	Coating Thickness and Condition					
	Substrate Material	Width, cm Depth, cm			1" from Fill Tube End Cap (Location "A" in Fig. 10)		Center (Location "B" in Fig. 10)		1" from Solid End Cap (Location "C" in Fig. 10)	
					Wall	Groove	Wall	Groove	Wall	Groove
1	Inconel 718	W: .102 D: .051	Aluminum Powder -40 Mesh 99.99% Pure Temperature 913K Duration 1000 Hours Orientation Horizontal Rate of Rotation 1 RPM	Na: 58 Al: 6	0.0023 cm	0.002 3cm	0.0079 cm	0.0023 cm	0.0079 cm	0.0033 cm
2	Inconel 718	W: .102 D: .102		Na: 58 Al: 6	Trace	0.0008 cm	0.0079 cm	0.0008 cm	0.0033 cm	0.0023 cm
3	Inconel 718	W: .102 D: .152		Na: 58 Al: 6	0.0048 cm	Essentially none	0.0079 cm	Trace	0.0064 cm	Trace
4	Inconel 718	W: .102 D: .051		Na: 58 Al: 18	0.0032 cm	Essentially none	Essentially none	Essentially none	0.0016 cm	Trace
5	Inconel 718	W: .102 D: .102		Na: 58 Al: 18	0.0064 cm	Essentially none	0.0111 cm	Essentially none	0.0079 cm	Trace
6	Inconel 718	W: .102 D: .152		Na: 58 Al: 18	Essentially none	0.0032 cm	0.0032 cm	Essentially none	0.0064 cm	Essentially none

0.152cm deep grooves. Based on work performed by Mr. Jack Devan who used lithium rather than sodium^[34,35] and the solubility of aluminum in sodium, the fluid charge for the pipes was 58g of sodium with 6g or 18g of aluminum powder. After charging, the pipes were processed at 913K to remove non-condensable gases. During testing, the pipes were operated at 913K (to remain below the aluminum melting point) for 1000 hours, oriented horizontally and rotated at one revolution per minute to uniformly wet the parallel groove specimens, and heated along the full length of the test pipe. After operation, the test vehicles were sectioned, photomicrographed and analyzed to determine the nature and suitability of the coating for groove wick structures. Figures 10 and 11 show a typical groove test pipe and parallel groove specimens, respectively. Note, samples were taken from the test pipe at the locations shown in Figure 10. Figure 12 shows the groove and screen test pipes during operation. Table 19 shows the test matrix and results for the groove test pipes. Selected photomicrographs for Groove Test Pipes Nos. 1 and 4 are included in Appendix M.

Although these tests indicated that aluminum doped sodium can be used to form nickel aluminide on the inside of fully-assembled heat pipes, the results were not uniform. In Groove Test Pipes Nos. 1 and 2, the coating was formed on the wall and in the grooves. In the remaining pipes, the coating was formed on the wall, but only a trace was formed in the grooves. Additional development work beyond the scope of this program is required to fully understand and develop this process.

2.3.2 Screen Wick Structures

The following sections detail nickel aluminide application processes evaluated for screen wick structures.

One-step and Two-step Nickel Aluminide Application Processes

For effective heat transfer in high heat flux applications, the wick structure must be in contact with the heat pipe wall. In this application, spot welding of the screen wick to the wall will assure effective heat transfer. Therefore, in addition to evaluating the ability to coat screen wicks with the one-step and two-step nickel aluminide processes, several test articles were also prepared to demonstrate spot welding of uncoated and coated screen wick samples to coated In718 substrates.

Using their two-step application process, Hitemco coated four In718 substrate specimens. The substrates were 5.1cm wide, 5.1cm long, and 0.64cm thick with a measured coating thickness of approximately 0.008cm. Two of the four nickel aluminide coated substrate specimens were used to evaluate Hitemco's coating. Visual inspection of these specimens indicated that the coating was similar to Chromalloy's coating used on the Phase I sample tubes. As a result, Thermacore believed that Hitemco's coating was acceptable for use on the Phase II sample tubes.

On two specimens, uncoated 100 mesh SS316 and Ni200 screens were successfully spot welded to nickel aluminide coated substrates. The screen samples were 2.5cm wide and 5.1cm long. One specimen was vacuum fired at 1073K for twenty-four hours. After firing, the screens were still attached to the coated substrate. Next, both specimens were sectioned, photomicrographed and analyzed. The photomicrographs indicated that a nickel aluminide layer of approximately 0.008cm was present on all In718 substrates. It should also be noted that the spot welds burn through the nickel aluminide coating.

Table 20. Test Matrix and Results for Nickel Aluminide Coated 100 and 60 Mesh Ni200 Screens.

Sample Number	Screen Mesh	Ni200 Screen Coating	In718 Substrate Coating	Fired at 1073K	Coating Thickness and Condition
1	100	One-step NiAl	Two-step NiAl	Yes 8 hr	0.0015 cm, no cracks
2	100	One-step NiAl	Two-step NiAl	No	0.0008 cm, no cracks
3	60	One-step NiAl	Two-step NiAl	Yes 8 hr	0.0008 cm, no cracks
4	60	One-step NiAl	Two-step NiAl	No	0.0008 cm, no cracks
5	60	Two-step NiAl	Two-step NiAl	Yes 8 hr	0.0023 cm, cracks
6	60	Two-step NiAl	Two-step NiAl	No	0.0023 cm, cracks
7	100	Two-step NiAl	Two-step NiAl	Yes 8 hr	0.0015 cm, cracks
8	100	Two-step NiAl	Two-step NiAl	No	0.0023 cm, cracks

Hitemco also coated two 100 mesh and two 60 mesh Ni200 screen samples using their one-step and two-step nickel aluminide application processes. On two Ni200 specimens, one 100 mesh and one 60 mesh, nickel aluminide coating was applied using the two-step nickel aluminide application process. During the first step, an essentially pure layer of aluminum was applied using a two hour, 1023K pack cementation process. These specimens were then heated for two hours at 1313K to diffuse the aluminum into the screen. Using this process, an average coating thickness of 0.0021cm was applied to the screen. On the remaining two Ni200 screen specimens, one 100 mesh and one 60 mesh, nickel aluminide coating was applied using a one-step, 1293K pack cementation process. Using this process, an average coating thickness of 0.0010cm was applied to the screen. For all screen samples, the wires and the points where one wire crosses over another had uniform layers of coating.

Hitemco coated eight In718 substrates using the two-step nickel aluminide process. Using this process, a coating thickness of approximately 0.013cm was formed on the substrates. Sections of each coated screen sample described above were spot welded to these nickel aluminide coated In718 substrates. Two specimens for each screen sample were prepared for evaluation. After the screens were attached to the substrates, one specimen for each sample was vacuum fired at 1073K for eight hours. After firing, the specimens were inspected to determine if the screen remained attached to the coated substrate. All screens remained attached to the substrates. The screens were then removed from both the unfired and fired substrates, sectioned, photomicrographed, and analyzed. The photomicrographs indicated that the one-step coating process produced coatings which did not crack and the two-step coating process produced coatings which did crack. As a result, the one-step process appears to be the better nickel aluminide process for coating screen wick structures. Table 20 shows the test matrix and results for this coating method. Figure 13 shows a coated screen specimen attached to a coated In718 substrate. Photomicrographs for these screen specimens are included in Appendix N.

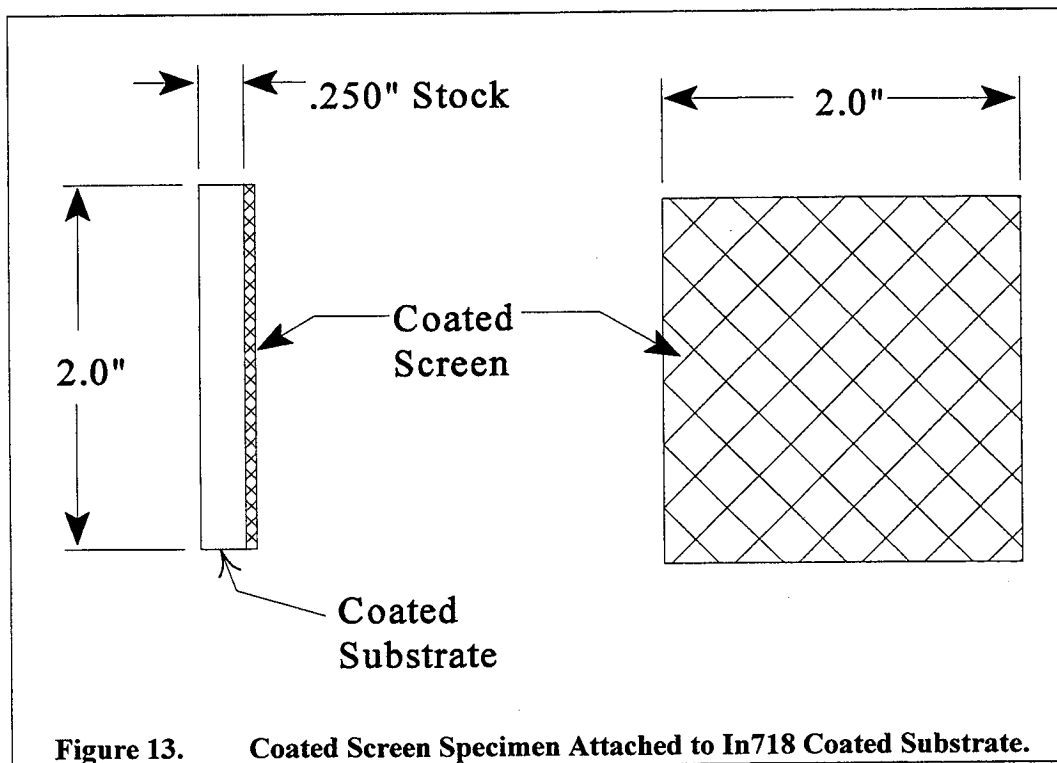


Figure 13. Coated Screen Specimen Attached to In718 Coated Substrate.

An attempt was also made to coat the screen and substrate after the screen was attached. Sections of uncoated 100 mesh and 60 mesh screen were spot welded to uncoated In718 substrates. Two specimens for each uncoated screen mesh were prepared. Hitemco coated these specimens with nickel aluminide using their one-step application process. After coating, one specimen for each screen mesh was vacuum fired at 1073K for eight hours. After firing, the specimens were inspected to determine if the screens remained attached to the coated substrates. All screens remained attached to the substrates. The screens were then removed from both the unfired and fired substrates, sectioned, photomicrographed and analyzed. The photomicrographs indicated that this coating process produced a coating which was thicker (0.0040cm) and had more cracks than the nickel aluminide coating applied to the screen which was then spot welded to nickel aluminide coated In718 substrates (discussed above). As a result, the screen and substrate should be nickel aluminide coated before the screen is spot welded to the substrate. Table 21 shows the test matrix and results for this coating method. Photomicrographs for these screen samples are included in Appendix O.

As an alternative to coating screen mesh, several tests were performed to demonstrate coating of Ni200 wire. After coating, the wire would be woven into screen mesh. Under this effort, Hitemco attempted to coat samples of 0.005cm and 0.015cm diameter Ni200 wires using the one-step nickel aluminide application process. After coating, both wire samples were brittle. The 0.005cm diameter wire fell apart when removed from the processing furnace. The 0.015cm diameter wire fell apart when prepared for photomicrography. Photomicrographs for the 0.005cm wire were not prepared. Photomicrographs for the 0.015cm wire are included in Appendix P. Since the nickel aluminide coated wires were extremely brittle, it does not appear feasible to weave screens using this wire.

Table 21. Test Matrix and Results for Nickel Aluminide Coated 100 and 60 Mesh Ni200 Screen.

Sample Number	Screen Mesh	Initial Ni200 Screen Coating	Initial In718 Substrate Coating	Coating Method	Fired at 1073K	Screen Coating Thickness and Condition
1	100	Uncoated	Uncoated	One-step NiAl	Yes 8 hr	0.0040cm; cracks
2	60	Uncoated	Uncoated	One-step NiAl	Yes 8 hr	0.0040cm; cracks
3	100	Uncoated	Uncoated	One-step NiAl	No	0.0040cm; cracks
4	60	Uncoated	Uncoated	One-step NiAl	No	0.0032cm; cracks

Two-step Aluminum to Nickel Aluminide Application Process

Titanium Finishing Company (TFC), East Greenville PA, vapor deposited a layer of aluminum onto 100 mesh and 60 mesh Ni200 screen specimens. Two samples of each screen mesh were then vacuum fired by Thermacore at 1293K for two hours to promote the formation of a nickel aluminide coating. These samples were then spot welded to two-step nickel aluminide coated In718 substrates. Two specimens, one 100 mesh and one 60 mesh, were vacuum fired at 1073K for eight hours. After firing, the specimens were inspected to determine if the screens remained attached to the substrates. All screens remained attached to the substrates. The screens were then removed from both the unfired and fired substrates, sectioned, photomicrographed and analyzed. Table 22 shows the test matrix and results for this coating method. Photomicrographs for samples 3 and 4 are included as Appendix Q.

Photomicrographs of the aluminum coated screen samples indicated that an average thickness of 0.0013cm of aluminum was uniformly deposited on the wires of the 60 mesh Ni200 screen, and an average thickness of 0.0018cm of aluminum was uniformly deposited on the wires of the 100 mesh Ni200 screen. Photomicrographs of the aluminum coated screen samples which were vacuum fired at 1293K for two hours indicated that an average thickness of 0.0013cm of nickel aluminide was uniformly formed on the wires of the 60 mesh Ni200 screen, and an average thickness of 0.0013cm of nickel aluminide was uniformly formed on the wires of the 100 mesh Ni200 screen. Photomicrographs of the aluminum coated screen samples which were vacuum fired at 1293K for two hours and then vacuum fired at 1073K for eight hours indicated that an average thickness of 0.0013cm of nickel aluminide was present on the wires of the 60 mesh Ni200 screen, and an average thickness of 0.0013cm of nickel aluminide was present on the wires of the 100 mesh Ni200 screen. The photomicrographs also indicated that the coatings on all screen samples were uniform and had no cracks.

TFC also vapor deposited a layer of aluminum onto 0.005cm and 0.015cm diameter Ni200 wire specimens. Four samples, two of each wire diameter, were vacuum fired at 1293K to promote the formation of a nickel aluminide coating. Two specimens, one 0.005cm diameter and one 0.015cm diameter, were then vacuum fired at 1073K for eight hours. After firing, the specimens were inspected to determine if the wires

Table 22. Test Matrix for Nickel Aluminide Coated Nickel 200 Screen Using the Two-Step Aluminum to Nickel Aluminide Application Process.

Sample Number	Screen Mesh	Initial Ni200 Screen Coating	In718 Substrate Coating	Firing Schedule	Coating Thickness and Condition
1	60	Vapor deposited aluminum	Two-step NiAl	None	0.0013 cm; no cracks
2	100	Vapor deposited aluminum	Two-step NiAl	None	0.0018 cm; no cracks
3	60	Vapor deposited aluminum	Two-step NiAl	1293K, two hours	0.0013 cm; no cracks
4	100	Vapor deposited aluminum	Two-step NiAl	1293K, two hours	0.0013 cm; no cracks
5	60	Vapor deposited aluminum	Two-step NiAl	1293K, two hours 1073K, eight hours	0.0013 cm; no cracks
6	100	Vapor deposited aluminum	Two-step NiAl	1293K, two hours 1073K, eight hours	0.0013 cm; no cracks

remained ductile. Both wires were more ductile than the coated wires prepared using the one-step nickel aluminide application process. The aluminum coated wires and the unfired and fired nickel aluminide coated wires were then sectioned, photomicrographed and analyzed. The photomicrographs were used to determine the coating's configuration and contact with the wire. Table 23 shows the test matrix and results for this coating method.

Photomicrographs of the aluminum coated wire samples indicated that an average thickness of 0.0008cm of aluminum was uniformly deposited on the 0.005cm diameter Ni200 wire, and an average thickness of 0.0013cm of aluminum was uniformly deposited on the 0.015cm diameter Ni200 wire. Photomicrographs of the aluminum coated wire samples which were vacuum fired at 1293K for two hours indicated that an average thickness of 0.0013cm of nickel aluminide was uniformly formed on the 0.005cm diameter Ni200 wire, and an average thickness of 0.0008cm of nickel aluminide was uniformly formed on the 0.015cm diameter Ni200 wire. Photomicrographs of the aluminum coated wire samples which were vacuum fired at 1293K for two hours and then vacuum fired at 1073K for eight hours indicated that an average thickness of 0.0008cm of nickel aluminide was present on the 0.005cm diameter Ni200 wire, and an average thickness of 0.0010cm of nickel aluminide was present on the 0.015cm diameter Ni200 wire. In addition, the photomicrographs indicated that the coatings on all wire samples had no cracks.

Since the nickel aluminide coating was uniformly formed on both the screen and wire samples, weaving 60 and 100 mesh screen from wire was not required. However, since the wires were brittle after firing, weaving 60 and 100 mesh screen would have been difficult.

Table 23. Test Matrix for Nickel Aluminide Coated Nickel 200 Wire Coated Using the Two-Step Aluminum to Nickel Aluminide Application Process.

Sample Number	Wire Dia.	Initial Ni200 Wire Coating	Firing Schedule	Coating Thickness and Condition
1	0.005cm	Vapor deposited aluminum	None	0.0008 cm; no cracks
2	0.015cm	Vapor deposited aluminum	None	0.0013 cm; no cracks
3	0.005cm	Vapor deposited aluminum	1293K, two hours	0.0013 cm; no cracks
4	0.015cm	Vapor deposited aluminum	1293K, two hours	0.0008 cm; no cracks
5	0.005cm	Vapor deposited aluminum	1293K, two hours 1073K, eight hours	0.0008 cm; no cracks
6	0.015cm	Vapor deposited aluminum	1293K, two hours 1073K eight hours	0.0010 cm; no cracks

Aluminum Doped Sodium Test Pipes for Coating Screen Specimens

As an alternative to forming the nickel aluminide coating on screen wick structures prior to installing the screen in the heat pipe, this method attempted to form a nickel aluminide coating on Ni200 screen wick structures within six closed SS316 test pipes containing an aluminum-doped sodium working fluid.

Two SS316 Pre-Matrix Screen Test Pipes were first operated to determine the optimum operating time for the doped Screen Test Pipes. Both test pipes had two layers of 60 mesh Ni200 screen. The fluid charge for the pipes was 29g of sodium with 3g of aluminum powder. After charging with sodium, the pipes were processed at 913K to remove non-condensable gases. During testing, the pipes were operated at 913K for 200 (#1) and 1000 (#2) hours, oriented horizontally and rotated at one revolution per minute to uniformly wet the screen wick structures, and heated along the full length of the test pipe. After operation, the test pipes were sectioned, photomicrographed and analyzed to determine the nature and suitability of the coating for the wall and screen wick structures. Figure 14 shows a typical screen test pipe. Figure 12 shows the groove and screen test pipes during operation. Table 24 shows the test matrix and results for the pre-matrix test pipes. After analyzing these pipes, 1000 hours was selected as the optimum operating time.

Six SS316 Screen Test Pipes were then operated to attempt to form nickel aluminide coating on Ni200 screen wick structures. Each Screen Test Pipe had two layers of 100 mesh or 60 mesh Ni200 screen. The fluid charge for the pipes was 58g of sodium with 6g, 12g, or 18g of aluminum powder. After charging with sodium, the pipes were processed at 913K to remove non-condensable gases. During testing, the pipes were operated at 913K for 1000 hours, oriented horizontally and rotated at one revolution per minute to uniformly wet the screen wick structure and heated along the full length of the test pipe. After operation, the test pipes were sectioned, photomicrographed and analyzed to determine the nature and suitability of the coating for the wall and screen wick structures. Table 25 shows the test matrix and results for the screen test pipes. Selected photomicrographs for Screen Test Pipes Nos. 1 and 3 are included in Appendix R.

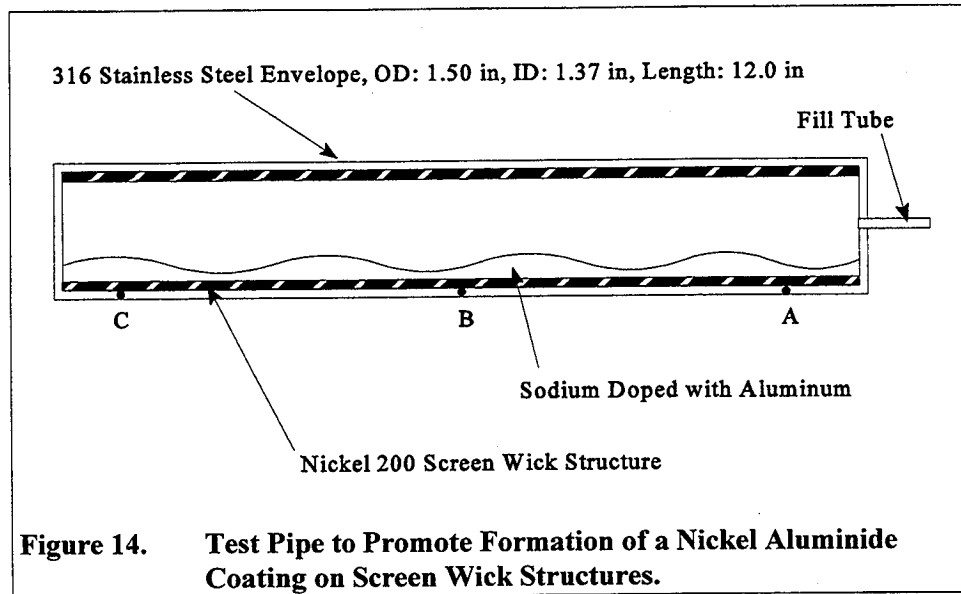


Table 24. Test Matrix and Results for the Task 3 Pre-Matrix Screen Test Pipes.

	Pre-Matrix Test Pipe #1	Pre-Matrix Test Pipe #2
SS 316 Pipe Diameter	2.5 cm	2.5 cm
SS316 Pipe Wall Thickness	0.165 cm	0.165 cm
SS316 Pipe Length	30.5 cm	30.5 cm
Wick Material	Two wraps, 60 mesh Ni200 screen	Two wraps, 60 mesh Ni200 screen
Operating Conditions	913K, 200 hours	913K, 1000 hours
Working Fluid	29.0 g Sodium, 3 g Aluminum	29.0 g Sodium, 3 g Aluminum
Wall Coating		
1" from fill tube end cap	Essentially no coating	Essentially no coating
Center of pipe	0.0008 cm	Essentially no coating
1" from solid end cap	0.0010 cm	0.0038 cm
Screen Coating		
1" from fill tube end cap	Essentially no coating	0.0008 cm
Center of pipe	Trace of coating	0.0008 cm
1" from solid end cap	0.0013 cm	0.0089 cm

The test pipes using sodium doped with aluminum powder produced coatings on the screen wick structures which were not uniform. In all Screen Test Pipes, the coating was formed on the screen (though non-uniform), but only a trace of coating was formed on some areas of the walls. Additional development work beyond the scope of this program is required to fully understand this process. Also, see the discussion in Section 2.4.1 on the successful test of a doped pipe with a screen wick structure that started with a nickel aluminide coating on the walls.

2.4 TASK 4: TEST OF CYLINDRICAL HEAT PIPES WITH COATINGS

The objective of Task 4 was to determine the durability and suitability of the Task 3 nickel aluminide coatings under heat pipe operating conditions. Cylindrical heat pipes were fabricated with wick structures coated using the best methods developed in Task 3.

2.4.1 Pre-matrix Screen Heat Pipes

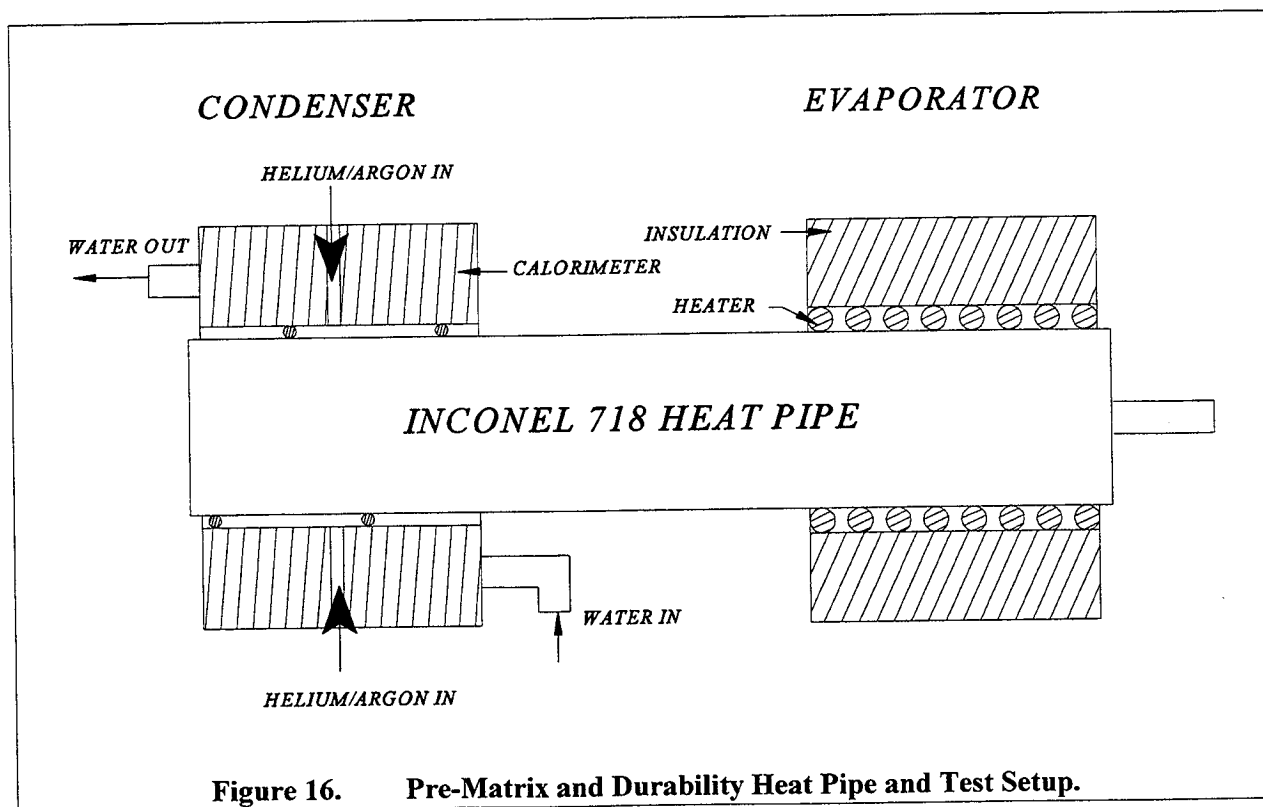
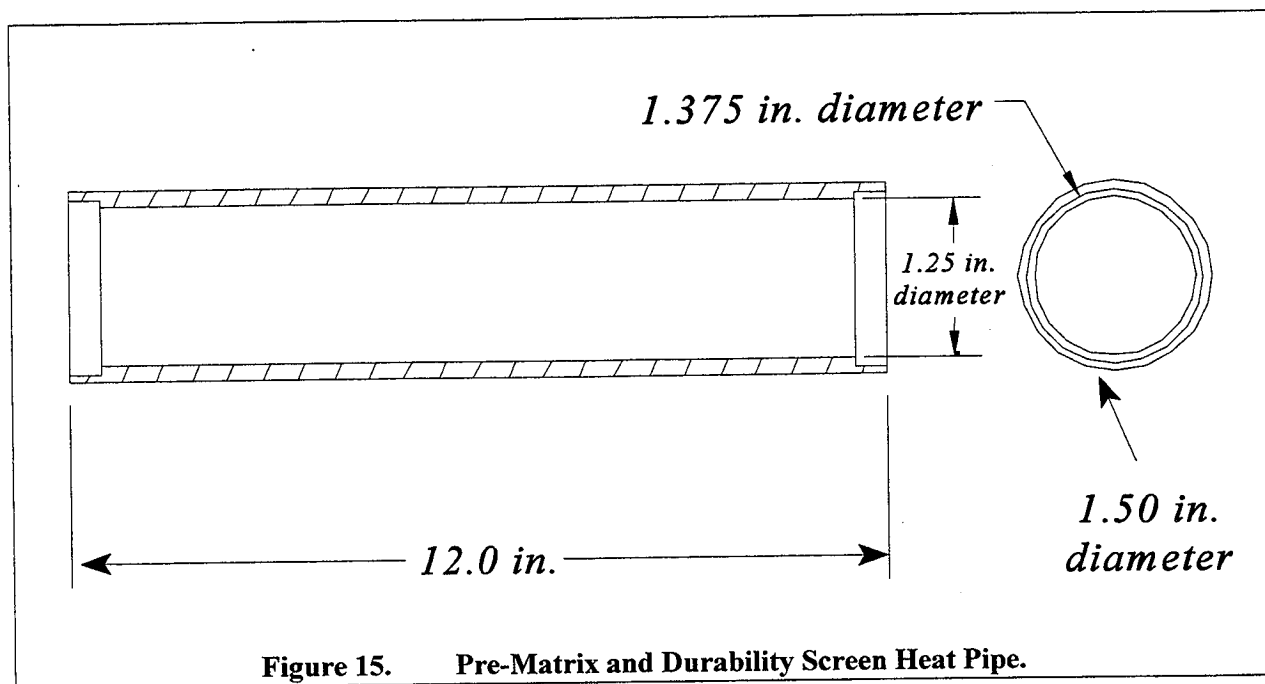
To expand upon the results from Task 3, three In718 cylindrical heat pipes were fabricated and operated for 1000 hours to determine the optimum method for applying a nickel aluminide coating to the wick structures for the Task 5 heat pipe. These pre-matrix heat pipes were started prior to the primary Task 4 5000-hour heat pipes discussed in Section 2.4.2 and were added to provide a timely input to the decision process for the Task 5 wick structure. Each heat pipe had two wraps of 100 mesh Ni200 screen. The heat pipe condensers were designed for a condensation heat flux of 25 W/cm^2 . Figures 15 and 16 show the design and test setup for the 1000 hour pre-matrix heat pipes. Table 26 shows the test matrix and results for the pre-matrix heat pipes. Selected post-test photomicrographs for the pre-matrix heat pipes are included in Appendix S.

Pre-Matrix Heat Pipe No. 1 was fabricated from In718 with nickel aluminide coated inner surfaces. Hitemco coated the inner envelope and end cap surfaces using their two-step nickel aluminide application process. Hitemco also coated the wick structure, two wraps of 100 mesh Ni200 screen, using their one-step nickel aluminide application process. The wick structure was installed in the coated heat pipe after the wick was coated. After fabrication, the pipe was charged with 58g of sodium, processed at 1073K to remove non-condensable gases and operated at 1073K for 1000 hours. During operation, the heat pipe was oriented with the condenser 0.64-1.27cm below the evaporator. After testing, the pipe was sectioned, photomicrographed and analyzed. Photomicrographs of these sections indicated that the evaporator screen was almost totally converted to nickel aluminide coating (wire diameter of 0.005 cm), approximately 0.0032cm of nickel aluminide coating was present on the condenser screen, approximately 0.0048cm of nickel aluminide was present on the on the evaporator wall, and approximately 0.0095cm of nickel aluminide was present on the condenser wall.

Pre-Matrix Heat Pipe No. 2 was fabricated from In718 with nickel aluminide coated inner surfaces. Hitemco coated the inner envelope and end cap surfaces using their two-step nickel aluminide application process. Titanium Finishing Company (TFC) vapor deposited aluminum onto the wick structure, two wraps of 100 mesh Ni200 screen. After coating, the screen was vacuum fired by Thermacore at 1293K for two hours to promote the formation of a nickel aluminide coating. The wick structure was installed in the coated heat pipe after the vacuum firing and consequent formation of the coating. After fabrication, the pipe was charged with 58g of sodium, processed at 1073K to remove non-condensable gases and operated at 1073K

Table 25. Test Matrix for Coating Screen Wick Structures in Test Pipes Using Sodium Doped with Aluminum Powder.

Test Pipe No.	Screen Specimen		Test Pipe Operating Conditions	Amount of Sodium, g and Aluminum, g	Coating Thickness and Condition					
	Wick Material	Number of Wraps			1" from Fill Tube End Cap (Location "A" in Fig. 14)		Center (Location "B" in Fig. 14)		1" from Solid End Cap (Location "C" in Fig. 14)	
					Wall	Screen	Wall	Screen	Wall	Screen
1	100 Mesh Ni200 (0.005 cm dia.)	2	Aluminum Powder -40 Mesh 99.99% Pure Temperature 913K Duration 1000 Hours Orientation Horizontal Rate of Rotation 1 RPM	Na: 58 Al: 6	Essentially none	0.0005 cm	Trace	0.0008 cm	0.0015 cm	0.0079 cm
2	100 Mesh Ni200	2		Na: 58 Al: 12	Essentially none	0.0008 cm	Essentially none	0.0008 cm	0.0095 cm	0.0032 cm
3	100 Mesh Ni200	2		Na: 58 Al: 18	Essentially none	0.0008 cm	Essentially none	0.0048 cm	0.0095 cm	Converted to NiAl
4	60 Mesh Ni200 (0.015 cm dia.)	2		Na: 58 Al: 6	Essentially none	0.0005 cm	0.0005cm	0.0023 cm	0.0008 cm	0.0041cm
5	60 Mesh Ni200	2		Na: 58 Al: 12	Essentially none	0.0008 cm	Essentially none	0.0008 cm	0.0048 cm	0.0079cm
6	60 Mesh Ni200	2		Na: 58 Al: 18	Essentially none	0.0008 cm	0.0064cm	0.0016 cm	0.0032 cm	0.0032cm



for 1000 hours. Photomicrographs of the sample sections indicated that only a trace of nickel aluminide coating remained on the evaporator screen, the condenser screen was almost totally converted to nickel aluminide, approximately 0.0079cm of nickel aluminide was present on the evaporator wall, and approximately 0.0064cm of nickel aluminide was present on the condenser wall.

Pre-Matrix Heat Pipe No. 3 was fabricated from In718 with nickel aluminide coated inner surfaces. Hitemco coated the inner envelope and end cap surfaces using their two-step nickel aluminide application process. The wick structure was two wraps of uncoated 100 mesh Ni200 screen. After fabrication, the pipe was charged with 58g of sodium doped with 18g of aluminum powder, processed at 913K to remove non-condensable gases, placed on a rotating test fixture and operated at 913K for 1000 hours. During operation, the pipe was oriented horizontally and rotated at one revolution per minute to uniformly wet the screen wick structure, and heated along the full length of the pipe. At the end of the coating process, the doped sodium was removed from the pipe. The heat pipe was then charged with 58g of sodium, processed at 1073K to remove non-condensable gases, operated at 1073K for 1000 hours and heated as shown in Figure 16. Photomicrographs of the sample sections indicated that approximately 0.0048cm of nickel aluminide coating was present on the evaporator screen, approximately 0.0048cm of nickel aluminide coating was present on the condenser screen, approximately 0.0064cm of nickel aluminide was present on the evaporator wall, and approximately 0.0079cm of nickel aluminide was present on the condenser wall.

Based on the results from the pre-matrix heat pipes, the one-step nickel aluminide coating process was selected for the Ni200 screen wick and artery structures and the two-step nickel aluminide process was selected for the In718 wall structure in the Task 5 heat pipe. In addition, no visible signs of corrosion were present in any of the pipes.

The one-step nickel aluminide process was selected despite some concern over the flexibility of the screen needed for the Task 5 wick installation. The two-step aluminum-to-nickel aluminide process gave a more flexible coating, but the lack of a reasonable coating on the evaporator screen of Pipe No. 2 at the end of the 1000 hour test was a significant concern.

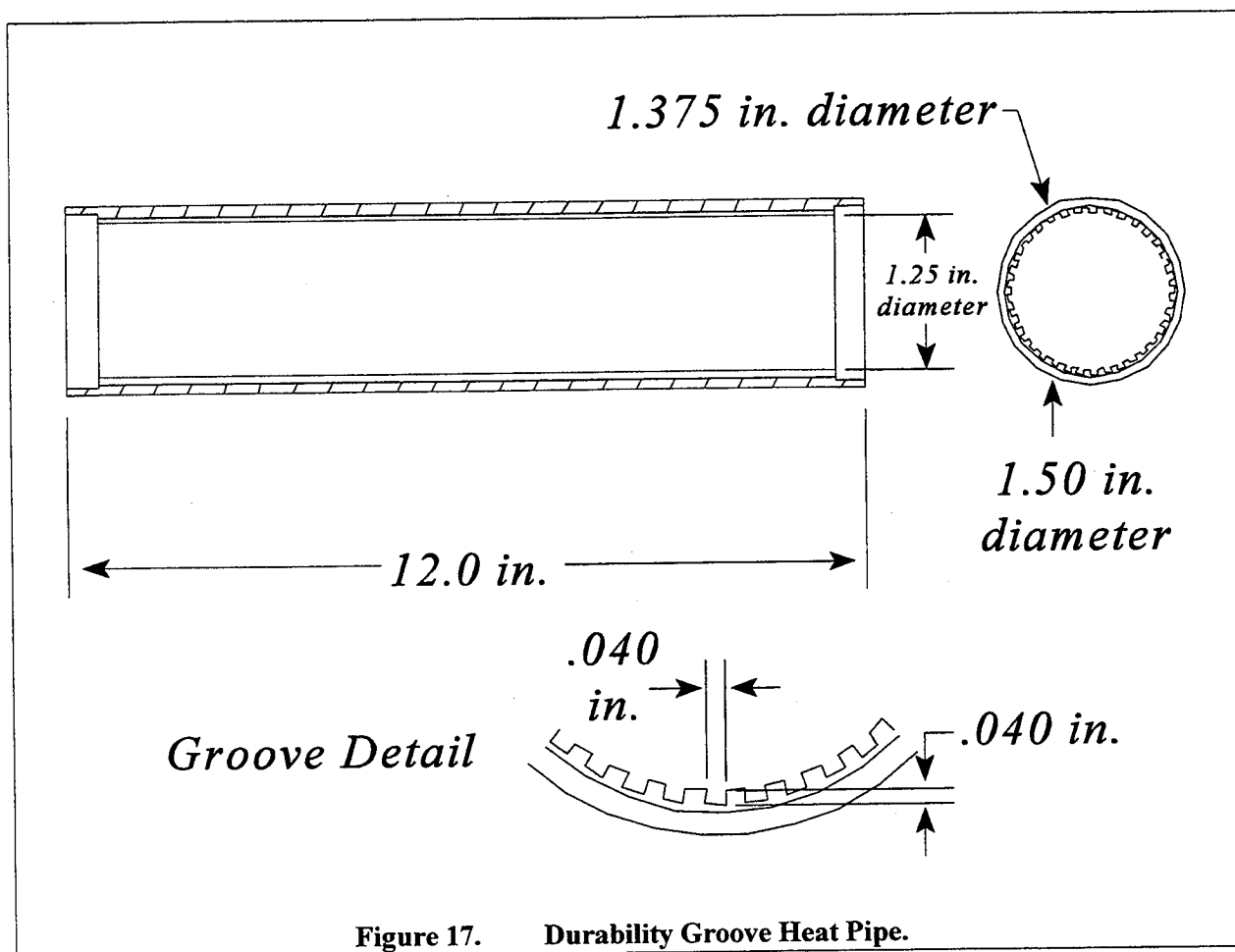
Also, note the successful results for doped Pre-Matrix Heat Pipe No. 3. The doped process had not been tried in Task 3 with a pipe wall that had been coated with nickel aluminide prior to attempting to form the screen coating by heating the doped fluid. These results were also measured after the additional 1000 hours of heat pipe operation which were not included in the Task 3 attempts. This process could not be pursued further due to the limited resources, but these results do provide encouragement for possible further efforts and potential success with this method.

2.4.2 Durability Heat Pipes

Five In718 cylindrical heat pipes were fabricated and four were operated to evaluate long life durability and suitability of the best coatings developed in Task 3. Four heat pipes had wick structures consisting of two wraps of 100 mesh Ni200 screen, and one heat pipe had a wick structure consisting of parallel grooves. For all pipes, the condenser end cap was first welded into the heat pipe envelope. For Durability Heat Pipe Nos. 1 through 3, Hitemco then coated the inner surfaces of this assembly and the evaporator end caps (except the surfaces which formed the evaporator-to-envelope weld joint) with nickel aluminide. The screen wick structures were then installed into Pipes Nos. 2 through 4 and the evaporator end caps welded into the envelopes. As a result, the condenser weld joint was coated and the evaporator weld joint was not coated.

Table 26. Test Matrix and Results for Task 4, 1000 Hour Pre-Matrix Screen Heat Pipes.

Heat Pipe No.	Sample Number	Sample Description	Coating Method	Operation Heat Flux, W/cm ²	Operating Conditions	Post-Test Coating Thickness and Condition
1 Figures 15 & 16	1	Middle of Evaporator: Wall	Two-step NiAl	5	Temperature: 1073K Duration: 1000 Hours Orientation: Condenser below evaporator Charge: 58g Na	0.0048 cm
	2	Middle of Evaporator: Screen	One-step NiAl	5		Conversion of screen to nickel aluminide.
	3	Middle of Condenser: Wall	Two-step NiAl	25		0.0095 cm
	4	Middle of Condenser: Screen	One-step NiAl	25		0.0032 cm
2 Figures 15 & 16	5	Middle of Evaporator: Wall	Two-step NiAl	5	Same as Pre-Matrix #1	0.0079 cm
	6	Middle of Evaporator: Screen	Two-step Al to NiAl	5		Trace of coating.
	7	Middle of Condenser: Wall	Two-step NiAl	25		0.0064 cm
	8	Middle of Condenser: Screen	Two-step Al to NiAl	25		Conversion of screen to nickel aluminide.
3 Figures 15 & 16	9	Middle of Evaporator: Wall	Two-step NiAl	5	A) Temperature: 913K Duration: 1000 hours Rotation: 1RPM Charge: 58g Na/18g Al B) Same as Pre-Matrix #1	0.0064 cm
	10	Middle of Evaporator: Screen	Na doped with 30 wt% Al	5		0.0048 cm
	11	Middle of Condenser: Wall	Two-step NiAl	25		0.0079 cm
	12	Middle of Condenser: Screen	Na doped with 30 wt% Al	25		Conversion of screen to nickel aluminide.



The heat pipe condensers were designed for a condensation heat flux of $25\text{W}/\text{cm}^2$. Figures 15, 16 and 17 show the design and test setup for the 5000 hour heat pipes. Table 27 shows the test matrix and results for the durability heat pipes. Selected post-test photomicrographs for the durability heat pipes are included in Appendix T.

Durability Heat Pipe No. 1 was fabricated from In718 with nickel aluminide coated inner surfaces. Hitemco coated the inner envelope and end cap surfaces using their two-step nickel aluminide application process. The inner wall included the 0.102cm deep, 0.102cm wide parallel groove wick structure. After fabrication, the pipe was charged with 58g of sodium, processed at 1073K to remove non-condensable gases and operated at 1073K for 5000 hours. During operation, the heat pipe was oriented with the condenser 0.64-1.27cm below the evaporator. After testing, the pipe was sectioned, photomicrographed and analyzed. The photomicrographs of the sample sections indicated that approximately 0.0095cm of nickel aluminide coating was present on the evaporator and condenser grooved wall surfaces. In addition, no signs of corrosion of the coating were visible.

Table 27. Test Matrix and Results for Task 4, 5000 Hour Screen Heat Pipes.

Heat Pipe No.	Sample No.	Sample Description	Coating Method	Heat Flux	Operating Conditions	Post-Test Coating Thickness and Condition
1 Figures 15 & 17	13	Middle of Evaporator: Grooved wall	Two-Step NiAl	5	Temperature: 1073K Duration: 5000 hrs Orientation: Condenser below Evaporator Charge: 58g Na	0.0095 cm; No corrosion
	15	Middle of Condenser: Grooved wall	Two-Step NiAl	25		0.0095 cm; No corrosion
2 Figures 15 & 16	17	Middle of Evaporator: Wall	Two-Step NiAl	5	Same as Durability # 1	0.0079 cm; No corrosion
	18	Middle of Evaporator: Screen	One-Step NiAl	5		0.0032 cm; No corrosion
	19	Middle of Condenser: Wall	Two-Step NiAl	25		0.0064 cm; No corrosion
	20	Middle of Condenser: Screen	One-Step NiAl	25		0.0032 cm; No corrosion
	21	Evaporator Weld Joint	Uncoated	N/A		No corrosion
	22	Condenser Weld Joint	Two-Step NiAl	N/A		No corrosion
3 Figures 15 & 16	23	Middle of Evaporator: Wall	Two-Step NiAl	5	Same as Durability # 1	0.0064 cm; No corrosion
	24	Middle of Evaporator: Screen	Two-Step Al to NiAl	5		0.0032 cm; No corrosion
	25	Middle of Condenser: Wall	Two-Step NiAl	25		0.0095cm; No corrosion
	26	Middle of Condenser: Screen	Two-Step Al to NiAl	25		Conversion of screen to nickel aluminide; No corrosion
4 Figures 15 & 16	27	Middle of Evaporator: Wall	Uncoated	5	Same as Durability # 1	No corrosion
	28	Middle of Evaporator: Screen	Uncoated	5		Corrosion of screen
	29	Middle of Condenser: Wall	Uncoated	25		No corrosion
	30	Middle of Condenser: Screen	Uncoated	25		Corrosion of screen
	31	Evaporator Weld Joint	Uncoated	N/A		No corrosion
	32	Condenser Weld Joint	Uncoated	N/A		No corrosion

Durability Heat Pipe No. 2 was fabricated from In718 with nickel aluminide coated inner surfaces. Hitemco coated the inner envelope and end cap surfaces using their two-step nickel aluminide application process. Hitemco also coated the wick structure, two wraps of 100 mesh Ni200 screen, using their one-step nickel aluminide application process. The wick structure was installed in the coated heat pipe after the wick was coated. After fabrication, the pipe was charged with 58g of sodium, processed at 1073K to remove non-condensable gases and operated at 1073K for 5000 hours. During operation, the heat pipe was oriented with the condenser 0.64-1.27cm below the evaporator. The post-test photomicrographs of the sample sections indicated that approximately 0.0032cm of nickel aluminide coating was present on the evaporator screen, approximately 0.0032cm of nickel aluminide was present on the condenser screen, approximately 0.0079cm of nickel aluminide was present on the evaporator wall, and approximately 0.0064cm of nickel aluminide was present on the condenser wall. In addition, no signs of corrosion of the coating, the evaporator weld joint, or the condenser weld joint were visible.

Durability Heat Pipe No. 3 was fabricated from In718 with nickel aluminide coated inner surfaces. Hitemco coated the inner envelope and end cap surfaces using their two-step nickel aluminide application process. TFC vapor deposited aluminum onto the wick structure, two wraps of 100 mesh Ni200 screen. After coating, the screen was vacuum fired by Thermacore at 1293K for two hours to promote the formation of a nickel aluminide coating. The wick structure was installed in the coated heat pipe after this vacuum firing and consequent formation of the coating. After fabrication, the pipe was charged with 58g of sodium, processed at 1073K to remove non-condensable gases and operated at 1073K for 5000 hours. During operation, the heat pipe was oriented with the condenser 0.64-1.27cm below the evaporator. The post-test photomicrographs of the sample sections indicated that approximately 0.0032cm of nickel aluminide coating was present on the evaporator screen, the condenser screen was almost totally converted to nickel aluminide (wire diameter of 0.005cm), approximately 0.0064cm of nickel aluminide was present on the evaporator wall, and approximately 0.0095cm of nickel aluminide was present on the condenser wall. In addition, no signs of corrosion of the coating were visible.

Durability Heat Pipe No. 4 (Control) was fabricated from In718 with uncoated inner surfaces. The wick structure was two wraps of uncoated 100 mesh Ni200 screen. After fabrication, the pipe was charged with 58g of sodium, processed at 1073K to remove non-condensable gases and operated at 1073K for 5000 hours. During operation, the heat pipe was oriented with the condenser 0.64-1.27cm below the evaporator. The post-test photomicrographs of the sample sections indicated that no signs of corrosion of the evaporator or condenser wall were visible, signs of moderate corrosion of the screen layers were visible, and no signs of corrosion of the evaporator or condenser weld joints were visible.

Durability Heat Pipe No. 5 was fabricated from In718 with nickel aluminide coated inner surfaces. Hitemco coated the inner envelope and end cap surfaces using their two-step nickel aluminide application process. In addition, the heat pipe wick structure was two wraps of uncoated 100 mesh Ni200 screen. The intent for this pipe was to use the doped sodium process to form the wick coating. However, the Task 3 results indicated that using aluminum-doped sodium heat pipes to promote the formation of a nickel aluminide coating on heat pipe wall and wick structures did not produce acceptable results. As a result, Thermacore and NASA LeRC agreed not to test Durability Heat Pipe No. 5. Note that the Task 4 Pre-Matrix Heat Pipe results for the doped pipe were found after this decision was made.

The results from the durability heat pipes supported the selection of the one-step nickel aluminide coating process for the Ni200 screen wick and artery structures and the two-step nickel aluminide for the In718 wall structure in the Task 5 heat pipe. In addition, the two-step aluminum-to-nickel aluminide process

gave very successful results and could also be used to reduce or prevent corrosion of wall and wick structures in sodium heat pipes. Note that there were no difficulties with the evaporator screen coating as seen in the pre-matrix heat pipe.

Finally, the results showed that there was no corrosion on the uncoated In718 heat pipe after this 5000 hour test, except for the uncoated Ni200 screen. The Task 2 final results, completed after these Task 4 results, definitely proved that SS316 (and not Ni200) should be used for uncoated screen wick structures.

2.5 TASK 5: FABRICATION AND DELIVERY OF A PARTIAL SEGMENT OF A HEAT PIPE FOR A STIRLING ENGINE POWER CONVERTER

As an example of a real world application, a full-scale, 1/10 segment of the current Stirling Space Power Converter (SSPC) Stirling engine heat pipe was fabricated and coated using the best processes developed in Tasks 2 through 4. Figures 18 and 19 show a photograph and an illustration of the 1/10 heat pipe during fabrication. The heat pipe was constructed from In718 and included a wick structure consisting of two layers of 100 mesh Ni200 screen and four, 200 mesh Ni200 screen arteries with 0.38cm outer diameter. One artery runs from each condenser well to the evaporator. The inner In718 surfaces were coated using the Hitemco two-step nickel aluminide application process discussed in Section 2.3. The Ni200 screen wick structure and arteries were coated using the two-step aluminum-to-nickel aluminide application process also discussed in Section 2.3 (originally attempted to coat the screen and arteries with the Hitemco one-step process - see discussion below). Table 28 lists the 1/10 segment heat pipe design specifications.

In the SSPC project, an uncoated 1/10 segment was built prior to fabricating the complete starfish heater and heat pipe to demonstrate the concept. The differences between the 1/10 segment heat pipe of this SBIR program (NASA LeRC NAS3-26925) and the partial segment heat pipe fabricated by Thermacore for the SSPC project under MTI Subcontract No. 003-05034 are:

- Nickel 200 screen replaces Stainless Steel 316 screen.
- Hitemco two-step nickel aluminide coating applied to heat pipe inner surfaces.
- Two-step aluminum-to-nickel aluminide coating applied to the screen wick and arteries.

The SSPC starfish heater consists of fifty radial fins with 38 one-millimeter diameter gas passages in each fin. The convertor's helium working fluid flows through the gas passages while the sodium in the heat pipe condenses on the outside of each fin. The annular heat pipe is attached to the outer radius of the starfish heater. The 1/10 segment is a 36° slice of the overall heater and heat pipe; the helium gas passages are replaced with slightly larger holes to allow installation of a calorimeter to remove and measure the heat flow. The starfish heater and heat pipe are discussed in References [2] and [3].

In January 1995, the layout and parts drawings for the 1/10 segment heat pipe were completed. In May 1995, machining of the heat pipe parts was completed. Following machining, the assembly (except for the top plate) was electron beam welded together. After these parts were welded, the screen wick, screen arteries, and the inner surfaces of the welded assembly were coated as described in the following paragraphs.

In August, Hitemco coated the inner wall surfaces of the heat pipe with their two-step nickel aluminide application process. During the months of August and September, Hitemco used a one-step nickel aluminide application process to coat the screen wick and artery structures for the 1/10 segment heat pipe

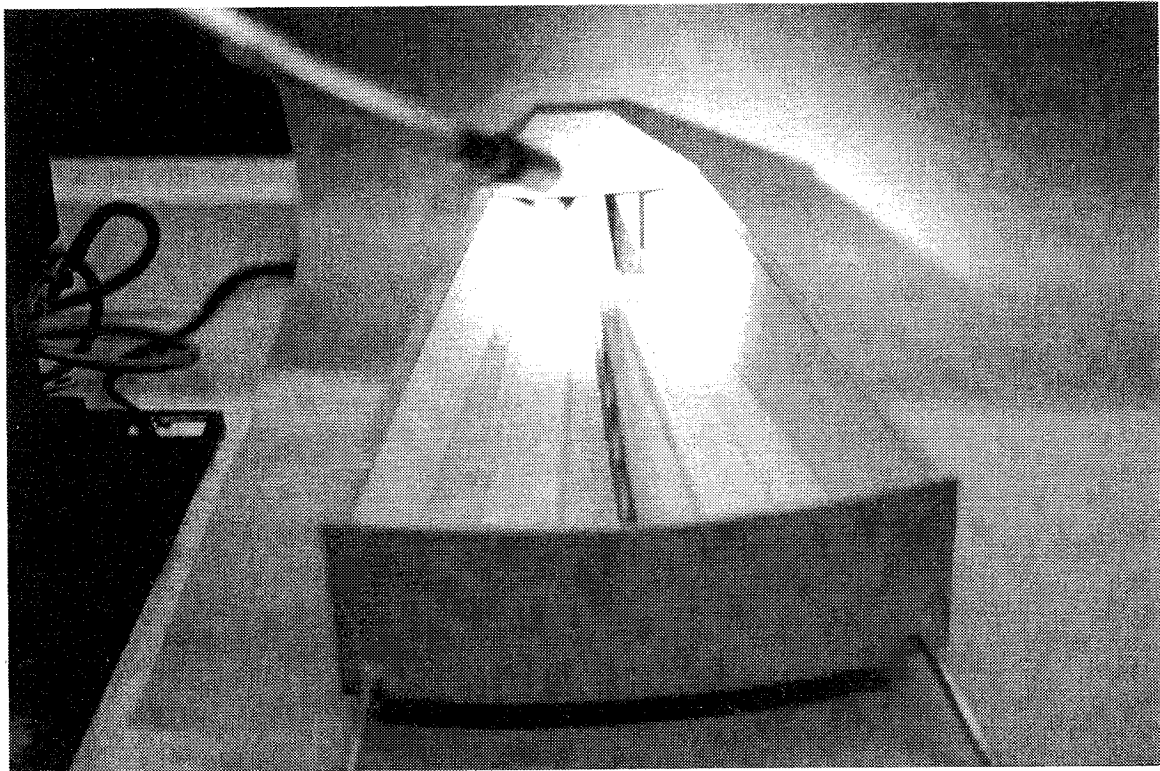


Figure 18. Coated Full-Scale, 1/10 Segment Heat Pipe without Top Plate.

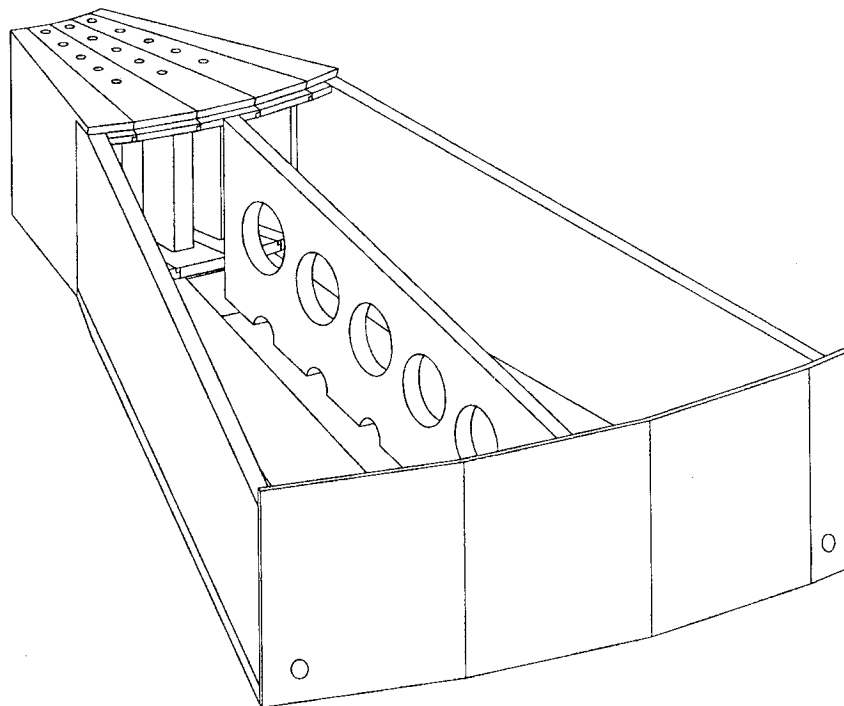


Figure 19. Full-Scale, 1/10 Segment Heat Pipe without Top Plate and Wick Structures.

Table 28. 1/10 Segment Heat Pipe Design Specifications.

PARAMETER	MAGNITUDE
Operating Temperature	1073K (+0/-20K)
Working Fluid and Fluid Charge	High Purity Sodium; 120g
Transport Capability	4500W
Condenser Heat Flux	20 W/cm ²
Heat Pipe Geometry	See Figures 18 and 19
Envelope Material and Fill Tube Material	Inconel 718 envelope; Stainless Steel 316L (fill tube)
Inner Surface Coating	Hitemco Two-Step NiAl Process
Wick Material	100 Mesh Nickel 200, Two Layers
Wick Coating	Two-Step Al (TFC)-to-NiAl Process
Number of Arteries	4
Artery Diameter	0.38cm Outer Dia.
Artery Material	200 Mesh Nickel 200, Two Layers
Artery Coating	Two-Step Al (TFC)-to-NiAl Process

assembly. The one-step coating process was the initial choice for coating the screen wick and artery structures based on the results of Task 3 and the Task 4 pre-matrix heat pipes. In October, Thermacore received the coated assembly and screen wick and artery structures. The coating process was performed twice for the inner wall surfaces, because the first attempt did not coat the top 2cm of the side walls.

In Task 3, the coated screen samples were spot welded onto flat uncoated and nickel aluminide coated In718 substrates. In Task 4, the coated screen wick structures were rolled and inserted into uncoated cylindrical In718 heat pipes. In these tasks, the screen was easily formed to these simple geometries. In this task, the geometry of the 1/10 heat pipe segment was more complicated and, as a result, forming the screen and attaching it to the heat pipe walls was more difficult. The difficulties encountered while attempting to spot weld two layers of one-step nickel aluminide coated Ni200 screen to the two-step nickel aluminide coated heat pipe assembly included the lack of a strong weld between the nickel aluminide coated Ni200 screen and the nickel aluminide coated In718 wall and cracking of the nickel aluminide coating on the wires of the Ni200 screen wires during welding.

In order to solve these problems, Thermacore evaluated a modification to the two-step aluminum-to-nickel aluminide process developed in Task 3 which would allow installing the Ni200 screen prior to the formation of the nickel aluminide. The idea was to install the screen after it was coated with aluminum and while it was still very flexible to allow better installation; the assembly would then be heated to form the nickel aluminide coating on the screen wick and artery structures. Thermacore performed tests which

included spot welding two layers of aluminum coated Ni200 screen to two, two-step nickel aluminide coated In718 substrates; vacuum firing the test articles at 1313K for two hours to promote a nickel aluminide coating on the screen; and analyzing the test articles. In both cases, the two screen layers remained strongly attached to the substrate. After the tests were completed, NASA LeRC and Thermacore decided to change coating methods for the screen wick and artery structures from the one-step coating process to the two-step aluminum-to-nickel aluminide coating process.

In January 1996, Thermacore received the aluminum-coated screen wick and artery structures. The first layer of aluminum coated screen was successfully spot welded into the two-step nickel aluminide coated heat pipe assembly and top plate. The artery structures were then located on the first layer of screen within the assembly. The second layer of screen was successfully spot welded into the assembly and onto the top plate. After the wick was installed, the heat pipe assembly and top plate were vacuum fired at 1313K for two hours to promote the formation of nickel aluminide on the screen wick structure.

As a check of the screen coating thickness, two layers of the aluminum coated screen were spot welded to a two-step nickel aluminide coated In718 substrate (5.1cm x 5.1cm). This sample was vacuum fired with the heat pipe assembly and the top plate to form the nickel aluminide coating. Analysis of the screen indicated that a 0.0008cm nickel aluminide coating thickness was formed.

In February, the top plate was electron beam welded onto the heat pipe assembly. After welding, the heat pipe was helium leak checked and found to be leak tight.

After fabrication and using Thermacore IR&D funding, the heat pipe was charged with high purity sodium, processed to remove non-condensable gases, and is scheduled to be life tested for up to ten years as a Phase III effort. During testing, the condenser section of the heat pipe will be coupled to a stainless steel calorimeter to measure heat rejection.

3.0 CONCLUSIONS AND RECOMMENDATIONS

The principal objective of this Phase II program was to develop and demonstrate a practically insoluble coating for nickel-based superalloys for Stirling engine heat pipe applications. Specific technical objectives of this program were:

- Determine the solubility corrosion rates for Nickel 200, Inconel 718 and Udimet 720LI in a simulated Stirling engine heat pipe environment,
- Develop coating processes and techniques for capillary groove and screen wick structures,
- Evaluate the durability and solubility corrosion rates for capillary groove and screen wick structures coated with an insoluble coating in cylindrical heat pipes operating under Stirling engine conditions,
- Design and fabricate a coated full-scale, partial segment of the current Stirling engine heat pipe for the Stirling Space Power Converter program.

Overall, the program was mostly successful and the stated goals were met. Based on the results of the Phase II program, specific conclusions and recommendations are listed below.

SOLUBILITY CORROSION TEST VEHICLES

1. After 2500 hours of testing in sodium at 1073K and a condensation heat flux of 25 W/cm², the analyses of the Ni200 test vehicle samples indicated an average corrosion rate of 0.0033cm/yr for the uncoated Ni200 sample tubes; an average corrosion rate of 0.0044cm/yr for the nickel aluminide coated Ni200 sample tubes (based on fewer samples and with much more deviation than for the uncoated samples); no signs of corrosion for the uncoated 100 mesh SS316 screen layers; and moderate deterioration for the uncoated 100 mesh Ni200 screen layers.
2. After 1886 hours of testing in sodium at 1073K and a condensation heat flux of 25 W/cm², the analyses of the Ud720LI test vehicle samples indicated no signs of corrosion for the uncoated Ud720LI sample tubes or for the nickel aluminide coated Ud720LI sample tubes. One of six photomicrographs showed corrosion for the uncoated 100 mesh SS316 screen layers while the other five showed no visible corrosion. The photomicrographs showed moderate corrosion for the uncoated 100 mesh Ni200 screen layers.
3. After 8767 hours of testing in sodium at 1073K and a condensation heat flux of 25 W/cm², the analyses of the In718 test vehicle samples indicated no signs of corrosion for the uncoated In718 sample tubes; essentially no signs of corrosion for the nickel aluminide coated In718 sample tubes; and no signs of corrosion for the uncoated 100 mesh SS316 screen layers. The uncoated 100 mesh Ni200 screen was reduced to dust.
4. Based on these results, the uncoated Ni200 screen should not be used for long term applications. Uncoated stainless steel screens showed little to no corrosion for any of the tests done in this project and, thus, should be a good choice for these applications. Coated Ni200 screen surfaces may be desired for very long life usage.

5. Based on these results and the Task 4 durability heat pipe results, there is no evidence that a heat pipe wall coating is needed for applications with lifetimes and operating conditions up to those studied in these tests. Coated surfaces may be desired for longer life and/or thin section, highly stressed parts.
6. Since the sample tubes became distorted during testing, the measurements (Appendix A procedure) of the sample tubes before and after testing have no reference from which to determine corrosion, and, thus, could not be used to determine corrosion rates.
7. For a majority of the sample tubes during testing, the plasma-sprayed tungsten and electroless plated nickel stripes remained attached to the nickel aluminide coating and the Ni200, In718 and Ud720LI substrates. In addition, diffusion of the stripe material into the coating or substrate was minimal. However, many reference stripes partially or totally disbonded from the coating or substrate during sectioning and preparation for photomicrography. The reference stripe technique worked well for determining corrosion when the stripe did not detach; a thinner reference stripe may make the stripe less susceptible to disbonding.
8. The solubility corrosion test vehicles design is reliable. However, extreme care must be used for joining dissimilar metals to the vessel.
9. The difficulty of determining corrosion from the solubility corrosion test vehicle samples should be noted. Maintaining an adequate reference line, interpreting the exact locations of the reference stripes and coating surfaces, and accounting for the tube curvature in some cases added to this difficulty.

HEAT PIPE WALL COATINGS

10. Hitemco's two-step nickel aluminide coating (a pack cementation process) was applied uniformly and consistently to In718, Ud720LI and Ni200 test samples and to In718 heat pipe wall surfaces. For up to 8767 hours on the In718 surfaces, the coating showed essentially no signs of corrosion in sodium at 1073K and a condensation heat flux of 25 W/cm². The coating also showed no signs of corrosion for up to 1886 hours on Ud720LI surfaces for the same temperature and heat flux.

PARALLEL GROOVE SUBSTRATE COATINGS

11. Two methods to apply nickel aluminide coatings to groove specimens were evaluated: a Hitemco two-step nickel aluminide application process using pack cementation and forming the coating in test pipes using sodium doped with aluminum powder.
12. Hitemco's two-step nickel aluminide coating was applied uniformly and consistently to In718 parallel groove wick samples with grooves of differing aspect ratios and to an In718 durability heat pipe with a groove wick structure. After 5000 hours of testing in the In718 heat pipe, the coating showed no signs of corrosion in sodium at 1073K and a condensation heat flux of 25 W/cm². Thermacore recommends using this two-step nickel aluminide application process for coating groove wick structures.

13. Aluminum powder doped sodium test pipes did not produce a uniform nickel aluminide coating on wall surfaces or groove wick structures. As a result, this process requires further development before it is feasible for coating wall or groove wick structures. Also, see statement 17 for further conclusions.

SCREEN WICK COATINGS

14. Three methods to apply nickel aluminide coating to screen specimens were evaluated: a two-step aluminum-to-nickel aluminide application process (aluminum vapor deposited by Titanium Finishing Company and then heat treated to form the nickel aluminide coating); Hitemco one-step and two-step nickel aluminide application processes (using pack cementation); and forming the coating in test pipes using sodium doped with aluminum powder.
15. The two-step aluminum-to-nickel aluminide coating was applied uniformly and consistently to various Ni200 screen wick structures, including an In718 durability heat pipe. After 5000 hours of testing in the In718 heat pipe, the coating showed no signs of corrosion in sodium at 1073K and a condensation heat flux of 25 W/cm². The two-step aluminum-to-nickel aluminide process produced coatings that were relatively ductile.

Thermacore recommends using the two-step aluminum-to-nickel aluminide application process for coating screen wick structures and especially for any applications that require the more ductile screen for installation purposes. To provide the most flexible screen for installation, aluminum should be applied to the screen wick structure before the wick is installed in the heat pipe. After installation of the wick structure, the wick is heat treated to diffuse the aluminum into the Ni200 and promote the formation of the nickel aluminide coating. The screen could be heat treated to form the nickel aluminide coating prior to the wick installation if further high temperature processing of the heat pipe materials is a concern. However, the screen is significantly less ductile after the nickel aluminide has formed.

16. Hitemco's one-step nickel aluminide coating was applied uniformly and consistently to various Ni200 screen wick structures, including an In718 durability heat pipe. After 5000 hours of testing in the In718 heat pipe, the coating showed no signs of corrosion in sodium at 1073K and a condensation heat flux of 25 W/cm². This coating should be applied to the screen wick structure before the wick is installed in the heat pipe. The one-step nickel aluminide process produced coatings that were more brittle than the two-step aluminum-to-nickel aluminide process. This one-step process should be acceptable for heat pipes with simpler screen installation and possibly where further high temperature processing of the heat pipe materials could lead to grain size and material property concerns.

The Hitemco two-step nickel aluminide process produced coatings on wire screens that were very brittle and were not considered satisfactory for screen wicking.

17. Aluminum powder doped sodium test pipes did not produce a uniform nickel aluminide coating on screen wick structures. As a result, this process requires further development before it is feasible for screen wick structures. However, the Task 4 test on a heat pipe that

started with coated walls and doped sodium did produce successful results and could serve as a starting point for further investigations. The doping process, if successful, could ensure coating of spot welds, screen crossovers, and otherwise uncoated close-out weld joints.

18. If the screen wick structure will be spot welded onto the sodium heat pipe walls, the two-step aluminum-to-nickel aluminide coating method is the most effective for attaching coated Ni200 screen wick structures.

DURABILITY HEAT PIPES

19. Successful 5000 hour testing of coated In718 durability heat pipes demonstrated no evidence of corrosion at the end of testing on wall surfaces coated with the two-step nickel aluminide process, grooves coated with the two-step nickel aluminide process, Ni200 screen wick structures coated with the one-step nickel aluminide process, and the Ni200 screen wick structures coated with the two-step aluminum-to-nickel aluminide process. The uncoated In718 control heat pipe also successfully completed the 5000-hour test with no evidence of corrosion on the wall surfaces but with moderate corrosion on the uncoated Ni200 screen.

FULL-SCALE, 1/10 SEGMENT SSPC HEAT PIPE

20. A coated full-scale, 1/10 segment heat pipe was fabricated to show the application of the nickel aluminide coating processes to the starfish heater / heat pipe geometry of the 12.5 kW_e Stirling Space Power Converter (SSPC). The two-step nickel aluminide process was used to coat the wall surfaces. The two-step aluminum-to-nickel aluminide process was used to coat the Ni200 screen wick structures and allow their successful installation. The additional high temperature processing steps necessary to apply the coating must be considered as they may affect material grain size and properties for the heat pipe wall surfaces.

4.0 REFERENCES

1. Mancini, T. R., Chavez, J. M., and Kolb, G. J. "Solar Thermal Power Today and Tomorrow." *Mechanical Engineering*, August 1994, pp. 74-79.
2. Dudenhoefer, J. E., Alger, D., and Winter, J. M. "Progress Update of NASA's Free-Piston Stirling Space Power Convertor Technology Project." NASA TM-105748, 1992.
3. Thieme, L. G. And Swec, D. M. "Summary of the NASA Lewis Component Technology Program for Stirling Power Convertors." NASA TM-105640, 1992.
4. Noble, J. E., Lehmann, G. A., and Emigh, S. G. "Materials for a Stirling Engine Heater Head." Proceedings of the 25th IECEC, Paper No. 900272, August 1990.
5. Johnson, R. N. "Tribological Coatings for Liquid Metal and Irradiation Environments," *Journal of Materials for Energy Systems*, Vol. 8 No. 1, pp. 27-37, June 1986.
6. Telephone conversation between James Lindemuth (Thermacore) and James Moreno and Charles Andraka (Sandia National Laboratories), May 2, 1994.
7. Charles E. Andraka, et. al. "NaK Pool-Boiler Bench-Scale Receiver Durability Test: Test Results and Materials Analysis." SAND94-1532C. Presented at Intersociety Energy Conversion Engineering Conference (IECEC 94-3865), August 1994.
8. Cavell, I.W. and Nicholas, M.G. "Some Effects of Exposing In718 and In718 Coated with Nickel Aluminide to Oxygenated Sodium." ETEC Library Reference No. LR-10141. United States Department of Energy-United Kingdom Atomic Energy Authority Fast Reactor Exchange Program, February 1979.
9. Johnson, R.N. "Nickel-Aluminide Diffusion Coating of Type 304 Stainless Steel Piston Rings for the Two-Stage Primary Pump Article." ETEC Library Reference No. LR-23482. Hanford Engineering Lab. for U.S. DOE, Sept. 1985.
10. Hammetter, F.W., Graham, R.A., Morosin, B. and Horie, Y. "Effects of Shock Modification on the Self-Propagating High Temperature Synthesis of Nickel Aluminides" in *Shock Waves in Condensed Matter 1987* (Schmidt, S. C. and Holmes, N. C., Editors). Proc. of the American Physical Society Topical Conference, Monterey CA, July 20-23, 1987.
11. Schindler-Latge, P., Ardellier, A. and Depierre, Y. "Sodium Compatibility of Aluminide and Chromium Nitride Coatings on Austenitic Stainless Steel Substrates." Proc. of the Fourth International Conference on Liquid Metal Engineering and Technology, Volume 2, Avignon, France, October 17-21, 1988.
12. "Titanium Aluminides: Tough Materials for Engines." *Mechanical Engineering*, Vol. 113, No. 12, December 1991.
13. Miller, L.G. and Beeston, J.M. "Extended Life Aluminide Fuel Final Report." EG&G Idaho, Inc.

for U.S. DOE, June 1986.

14. Lai, G.Y. "Investigation of Several Commercial Aluminide Coatings for Carburization Protection of a Nickel-Base Alloy." General Atomic Company for the San Francisco Operations Office - Department of Energy, June 1980.
15. Van Vlack, L. *Material Sciences for Engineers*, Addison-Wesley Publishing Co., Reading MA, 1970.
16. *Smithells Metals Reference Book*, 6th Edition. Eric A. Brandes, Editor. Butterworths, London, pp. 14-2 to 14-5, 1983
17. *Metals Handbook*, Volume 2, Properties and Selection: Nonferrous Alloys and Special Purpose Materials, 10th Edition, ASM International, 1990, pp. 687, 694, 806, 807, 1150, 1153.
18. *CRC Handbook of Chemistry and Physics*. Robert C. Weast, Ph.D., Editor-in-Chief, 64th Ed., Boca Raton, FL, p. F-47, F-49, 1984.
19. *Liquid Metals Handbook*. Richard N. Lyons, Editor-in-Chief. Atomic Energy Commission, pp. 147, 152, 1952.
20. "Zircadyne Corrosion Properties." Teledyne Wah Chang Albany. TWCA 8614ZR, p. 12, 1991.
21. *Metals Handbook*, Volume 13, Corrosion, 9th Edition. ASM International, pp. 717, 1987.
22. "Hafnium." Teledyne Wah Chang Albany. P. 14, 1987.
23. *Aerospace Structural Materials Handbook*, 1987 Edition, Volume 5, Code 4207, pp. 1-41.
24. *Aerospace Structural Metals Handbook*, 1987 Edition, Volume 4, Code 4103, pp. 2, 11.
25. "Periodic Table of the Elements," Sargent-Welch Scientific Company, Skokie, Illinois, 1979.
26. Mittendorf, D.L. and W.G. Baggenstoss. "Transient Liquid Phase Diffusion Bonding of Udimet 720 for Stirling Power Converter Applications." Proc. of the 27th IECEC, pp. 5.393 - 5.397, San Diego, CA, August 1992.
27. Baggenstoss, W. and D. Mittendorf. "Materials Technology for Stirling Space Power Converters." NASA Contractor Report 189102, July 1992.
28. Thieme, L.G. and D.M. Swec. "Overview of the NASA Lewis Component Technology Program for Stirling Power Converters." Proc. of the 27th IECEC, pp. 5.283 - 5.288, San Diego, CA, August 1992.
29. Private communication between James E. Lindemuth (Thermacore, Inc.) and Manmohan Dhar (Mechanical Technology Incorporated) on October 27, 1993.

30. From faxes provided by AAA Metals Company, Inc., 68 Industrial Boulevard, Hanson, MA 02341. PH: 800-531-9500.
31. *Aerospace Structural Materials Handbook*, 1987 Edition, Vol. 5, Code 4103, pp. 1-58.
32. *Aerospace Structural Materials Handbook*, 1987 Edition, Vol. 5, Code 4215, pp. 1-12.
33. *Aerospace Structural Materials Handbook*, 1987 Edition, Vol. 5, Code 4214, pp. 1-18.
34. DeVan, J. H. and J. E. Selle. "Method for Inhibiting Alkali Metal Corrosion of Nickel Containing Alloys." U. S. Patent No. 4,398,967, August 1983.
35. DeVan, J. H. "Corrosion of Iron- and Nickel-Based Alloys in High-Temperature Sodium and NaK." Work Sponsored by U. S. Atomic Energy Commission under Contract with Union Carbide Corporation.
36. McGuire, J. C. and W. F. Brehm. "Hydrogen Permeation Resistant Barrier." U. S. Patent No. 4,314,880, February 1982.

APPENDIX A

PROCEDURE TO MEASURE NICKEL 200, INCONEL 718 AND UDIMET 720LI SAMPLE TUBES

1. The following equipment is required:

- A. Inspection room at Thermacore's sister company Dynatherm Inc., Cockeysville, Maryland. Temperature: 70-75 °F; Relative Humidity: <50%.
- B. Brown & Sharpe BesTest Dial Indicator Model No. 599-7033-5, 0-0.008in range, 0.00005in graduations, 0-4-0 dial reading, 1.5 inch diameter dial, black dial face (available from McMaster-Carr).
- C. Dial Indicator Stand and Holder.

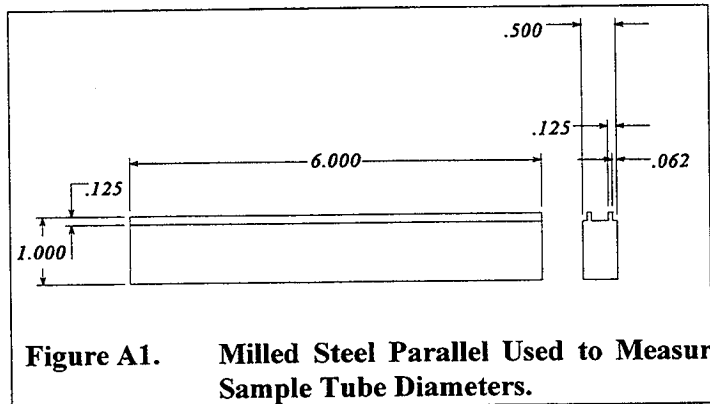
- D. Steel Parallel ($\frac{1}{2}$ " x 1" x 6") milled to the dimensions shown in Figure A1.

- E. Flat Granite Inspection Block.

- F. Micrometer.

- G. Pin Gauges.

- H. Uncoated Sample Tubes.
Store tubes in inspection room at least twelve hours before measurements are taken.



- 1. Nine Ni200 Sample Tubes A-11-1230-23 and A-11-1230-25.
- 2. Eight In718 Sample Tubes A-11-1230-24 and A-11-1230-25.
- 3. Nine Ud720LI Sample Tubes A-11-1230-24 and A-11-1230-25.

I. Sample Tube Data Sheets.

- 2. Select the Ni200 sample tubes (1.H.1).
- 3. Assign each tube a unique number from 1 to 9.
- 4. Select the In718 sample tubes (1.H.2).
- 5. Assign each tube a unique number from 10 to 17.
- 6. Select the Ud720LI sample tubes (1.H.3).
- 7. Assign each tube a unique number from 18 to 26.
- 8. Record the information from Steps 2 through 7 on the Sample Tube Data Sheets.

9. Select Tube 1.
10. Using the micrometer, measure the tube diameter.
11. Select a pin gauge which corresponds to the diameter measured in Step 10. Record the pin gauge diameter on the Sample Tube Data Sheet.
12. Set up dial indicator, dial indicator stand and holder, and V-block as shown in Figure A2.
13. Place selected pin gauge in the milled parallel as shown in Figure A2.
14. Zero the dial indicator using the selected pin gauge. After zeroing, place a check mark in the box labeled "Zero" on the Sample Tube Data Sheet.
15. Remove pin gauge.

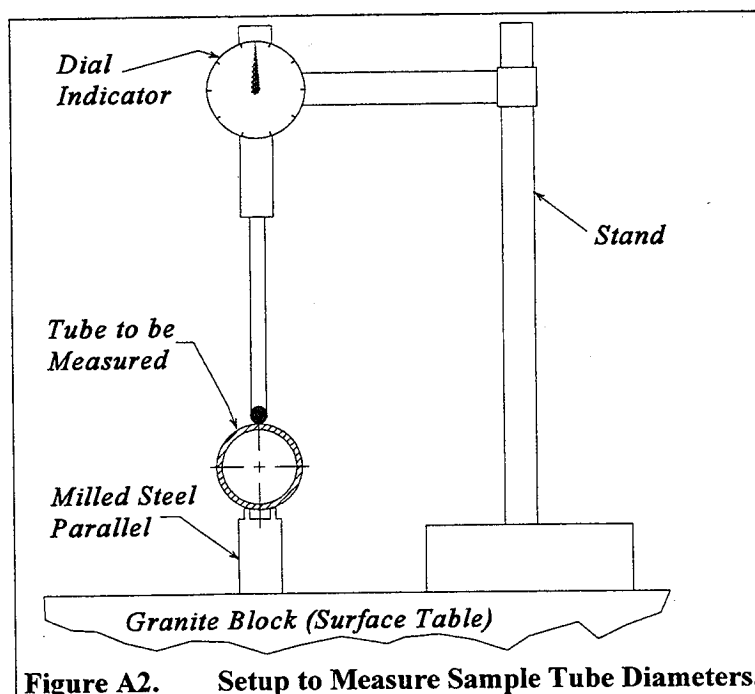


Figure A2. Setup to Measure Sample Tube Diameters.

16. Place tube in the milled parallel as shown in Figure A2.
17. Using the dial indicator, measure the tube diameter at the locations shown in drawing A-11-1230-21 (See Figure A3). Record the tube measurements on the Sample Tube Data Sheet.
18. Remove tube.
19. Place selected pin gauge in the milled parallel as shown in Figure A3. Dial indicator should read zero. If so, then place a second check mark in the box labeled "Zero" on the Sample Tube Data Sheet. If not, then repeat Steps 10 through 19 for present tube.
20. Repeat Steps 10 through 19 for Tubes 2 through 26.
21. Complete Sample Tube Data Sheet for Tubes 1 through 26.

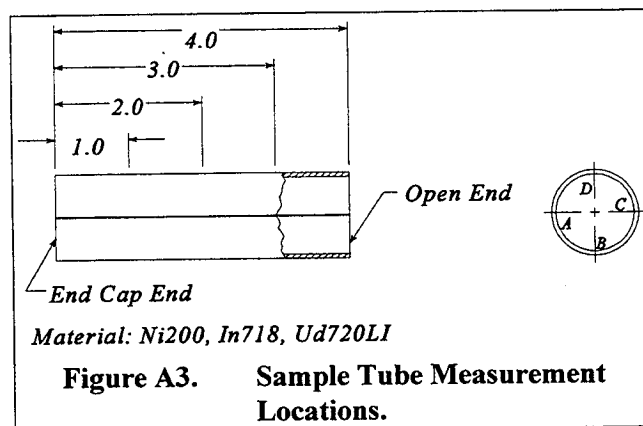


Figure A3. Sample Tube Measurement Locations.

APPENDIX B

**MEASUREMENTS FOR COATED AND UNCOATED NICKEL 200, INCONEL 718,
AND UDIMET 720 SAMPLE TUBES BEFORE AND AFTER TESTING**

Table B1. Measurement Data for Uncoated Nickel 200, Inconel 718 and Udimet 720 Sample Tubes Before Testing.

Sample Tube		Pin Guage (Inch)	Zero	At 1.0 Inch Mark (inches)				At 2.0 Inch Mark (inches)				At 3.0 Inch Mark (inches)				Coating Material	Screen Material	Tube Use
Material	Number			A	B	C	D	A	B	C	D	A	B	C	D			
12-13-93 Uncoated Ni200 and Inconel 718 sample tubes dropped off at Dynatherm.																		
12-13-93, 4:00 PM. Temperature: 294.5 K (70.6 F). Relative Humidity: --. JEL, GOB.																		
12-14-93, 8:30 AM. Temperature: 293.9 K (69.6 F). Relative Humidity: --																		
12-14-93, 10:00 AM. Temperature: 294.9 K (71.4 F). Relative Humidity: 36%																		
Nickel	1	.9855	Y/Y	.98620	.98715	.98715	.98680	.98620	.98700	.98715	.98695	.98680	.98700	.98695	.98680	NiAl	None	Test: 230hr
Nickel	2	.9855	Y/Y	.98680	.98745	.98710	.98735	.98730	.98775	.98710	.98720	.98760	.98765	.98700	.98700	NiAl	None	Test: 2270hr
Nickel	3	.9855	Y/Y	.98675	.98720	.98695	.98730	.98720	.98670	.98720	.98725	.98670	.98670	.98660	.98710	NiAl	SS316	Test: 2500hr
Nickel	4	.9855	Y/Y	.98680	.98660	.98700	.98765	.98655	.98665	.98705	.98750	.98670	.98665	.98670	.98670	NiAl	Ni200	Test: 230hr
Nickel	5	.9855	Y/Y	.98710	.98695	.98720	.98705	.98680	.98685	.98720	.98660	.98695	.98690	.98700	.98690	NiAl	Ni200	Test: 2270hr
Nickel	6	.9855	Y/Y	.98680	.98640	.98790	.98730	.98700	.98690	.98745	.98715	.98705	.98695	.98685	.98675	None	None	Specimen
12-14-93, 12:00 PM. Temperature: 295.4 K (72.4 F). Relative Humidity: 41%.																		
Nickel	7	.9855	Y/Y	.98655	.98690	.98745	.98750	.98670	.98690	.98710	.98695	.98660	.98695	.98690	.98680	None	None	Test: 2500hr
Nickel	8	.9855	Y/Y	.98620	.98685	.98665	.98675	.98675	.98635	.98680	.98680	.98700	.98655	.98690	.98700	None	SS316	Test: 2500hr
Nickel	9	.9855	Y/Y	.98695	.98660	.98710	.98730	.98660	.98685	.98715	.98730	.98680	.98680	.98660	.98675	None	Ni200	Test: 2500hr
12-14-93, 12:30 PM. Temperature: 295.7 K (72.8 F). Relative Humidity: 41%.																		
12-14-93, 1:00 PM. Temperature: 295.6 K (72.6 F). Relative Humidity: 40%.																		
Inconel	10	.9855	Y/Y	.98605	.98690	.98635	.98675	.98640	.98720	.98635	.98690	.98665	.98735	.98665	.98720	NiAl	None	Test
Inconel	11	.9855	Y/Y	.98655	.98655	.98640	.98660	.98685	.98655	.98645	.98655	.98700	.98665	.98670	.98665	NiAl	SS316	Test
Inconel	12	.9855	Y/Y	.98615	.98660	.98620	.98640	.98600	.98670	.98630	.98650	.98620	.98700	.98650	.98680	NiAl	Ni200	Test
Inconel	13	.9855	Y/Y	.98600	.98680	.98625	.98685	.98640	.98705	.98640	.98690	.98680	.98715	.98670	.98700	NiAl	Ni200	Specimen
Inconel	14	.9855	Y/Y	.98650	.98650	.98655	.98640	.98630	.98675	.98645	.98660	.98630	.98710	.98650	.98710	None	None	Test
Inconel	15	.9855	Y/Y	.98670	.98575	.98675	.98610	.98675	.98600	.98675	.98630	.98685	.98625	.98685	.98645	None	None	Specimen
Inconel	16	.9855	Y/Y	.98650	.98620	.98640	.98640	.98635	.98650	.98665	.98650	.98650	.98685	.98670	.98680	None	SS316	Test
Inconel	17	.9855	Y/Y	.98640	.98650	.98645	.98645	.98640	.98690	.98620	.98670	.98655	.98705	.98635	.98695	None	Ni200	Test
12-14-93, 1:30 PM. Temperature: 296.3 K (74.0 F). Relative Humidity: 40%.																		
3-17-94 Uncoated Ud720Li sample tubes dropped off at Dynatherm.																		
3-23-94, 10:40 AM. Temperature: 297.4 K (76.0 F). Relative Humidity: 40%. JEL, GOB.																		
Udimet	18	.9890	Y/Y	.98965	.98970	.98965	.98970	.98965	.98965	.98965	.98965	.98950	.98955	.98955	.98955	NiAl	SS316	Specimen
Udimet	19	.9890	Y/Y	.98980	.98970	.98970	.98970	.98980	.98975	.98970	.98975	.98965	.98970	.98970	.98965	NiAl	None	Test
Udimet	20	.9895	Y/Y	.98970	.98960	.98965	.98960	.98970	.98965	.98965	.98970	.98965	.98960	.98970	.98960	NiAl	Ni200	Specimen
Udimet	21	.9895	Y/Y	.98945	.98950	.98950	.98950	.98960	.98960	.98955	.98960	.98950	.98950	.98950	.98955	NiAl	SS316	Test
Udimet	22	.9895	Y/Y	.98930	.98940	.98940	.98940	.98940	.98940	.98940	.98940	.98925	.98925	.98925	.98930	NiAl	Ni200	Test
3-23-94, 11:40 AM. Temperature: 297.4 K (76.0 F). Relative Humidity: 40%.																		
Udimet	23	.9895	Y/Y	.98955	.98945	.98955	.98950	.98955	.98960	.98955	.98955	.98950	.98950	.98950	.98950	None	None	Test
Udimet	24	.9895	Y/Y	.98945	.98950	.98945	.98960	.98955	.98960	.98955	.98955	.98950	.98950	.98950	.98950	None	SS316	Test
Udimet	25	.9895	Y/Y	.98935	.98950	.98935	.98940	.98945	.98945	.98945	.98950	.98940	.98940	.98940	.98945	None	Ni200	Test
Udimet	26	.9895	Y/Y	.98970	.98955	.98960	.98955	.98960	.98960	.98965	.98965	.98960	.98955	.98960	.98960	None	None	Specimen
3-23-94, 11:50 AM. Temperature: 297.4 K (76.0 F). Relative Humidity: 40%.																		

Table B2. Measurement Data for Coated Nickel 200, Inconel 718 and Udimet 720 Sample Tubes Before Testing.

Sample Tube		Pin Gauge (Inch)	Zero	At 1.0 Inch Mark (inches)				At 2.0 Inch Mark (inches)				At 3.0 Inch Mark (inches)				Coating Material	Screen Material	Tube Use
Material	Number			A	B	C	D	A	B	C	D	A	B	C	D			
2-15-94 Coated Ni200 and In718 sample tubes dropped off at Dynatherm.																		
2-16-94, 11:50 AM. Temperature: 299.0 K (78.0 F). Relative Humidity: 36%. JEL, GOB.																		
Nickel	1	.9860	Y/Y	.98615	.98620	.98610	.98615	.98605	.98610	.98600	.98600	.98595	.98595	.98590	.98595	NiAl	None	Test: 230hr
Nickel	2	.9860	Y/Y	.98615	.98620	.98620	.98625	.98610	.98615	.98610	.98620	.98605	.98615	.98610	.98610	NiAl	None	Test: 2270hr
Nickel	3	.9860	Y/Y	.98615	.98625	.98620	.98620	.98615	.98615	.98615	.98615	.98615	.98615	.98610	.98610	NiAl	SS316	Test: 2500hr
Nickel	4	.9860	Y/Y	.98620	.98620	.98625	.98620	.98620	.98615	.98620	.98615	.98620	.98615	.98615	.98620	NiAl	Ni200	Test: 230hr
Nickel	5	.9860	Y/Y	.98610	.98610	.98605	.98615	.98600	.98610	.98600	.98605	.98595	.98605	.98600	.98605	NiAl	Ni200	Test: 2270hr
2-16-94, 12:45 PM. Temperature: 299.0 K (78.0 F). Relative Humidity: 36%.																		
Inconel	10	.9860	Y/Y	.98595	.98595	.98600	.98600	.98600	.98605	.98605	.98600	.98605	.98615	.98610	.98605	NiAl	None	Test
Inconel	11	.9860	Y/Y	.98630	.98635	.98635	.98635	.98635	.98635	.98635	.98640	.98640	.98640	.98635	.98640	NiAl	SS316	Test
Inconel	12	.9860	Y/Y	.98610	.98615	.98615	.98610	.98625	.98630	.98630	.98625	.98635	.98640	.98640	.98645	NiAl	Ni200	Test
Inconel	13	.9860	Y/Y	.98630	.98630	.98625	.98630	.98630	.98625	.98625	.98630	.98620	.98630	.98625	.98625	NiAl	Ni200	Specimen
2-16-94, 1:00 PM. Temperature: 299.0 K (78.0 F). Relative Humidity: 36%.																		
4-25-94 Coated Ud720LI sample tubes dropped off at Dynatherm.																		
4-26-94, 9:45 AM. Temperature: 297.4 K (76.0 F). Relative Humidity: 38%. JEL, GOB.																		
Udimet	18	.9870	Y/Y	.98760	.98720	.98740	.98720	.98750	.98740	.98735	.98775	.98735	.98740	.98750	.98750	NiAl	SS316	Specimen
Udimet	19	.9870	Y/Y	.98750	.98750	.98745	.98750	.98755	.98760	.98740	.98780	.98750	.98755	.98735	.98770	NiAl	None	Test
Udimet	20	.9870	Y/Y	.98740	.98755	.98740	.98750	.98790	.98765	.98760	.98725	.98755	.98765	.98730	.98745	NiAl	Ni200	Specimen
Udimet	21	.9870	Y/Y	.98750	.98740	.98750	.98735	.98760	.98745	.98750	.98755	.98760	.98725	.98750	.98730	NiAl	SS316	Test
Udimet	22	.9870	Y/Y	.98730	.98765	.98725	.98745	.98765	.98755	.98740	.98735	.98750	.98740	.98740	.98725	NiAl	Ni200	Test
4-26-94, 10:20 AM. Temperature: 297.4 K (76.0 F). Relative Humidity: 40%.																		

Table B3. Measurement Data for Uncoated and Coated Nickel 200 Sample Tubes After Testing.

Sample Tube		Pin Gauge (Inch)	Zero	At 1.0 Inch Mark (inches)				At 2.0 Inch Mark (inches)				At 3.0 Inch Mark (inches)				Coating Material	Screen Material	Tube Use
Material	Number			A	B	C	D	A	B	C	D	A	B	C	D			
1-23-95 Uncoated and Coated Ni200 sample tubes placed in Quality Assurance room at Thermacore.																		
1-24-95, 8:57 AM. Temperature: 294.2 K (70.1 F). Relative Humidity: 43%. JEL, GOB.																		
Nickel	1	.9885	Y/Y	.98810	.98670	.99290	.98580	.99420	.98580	.99135	.98580	.98875	.98740	.98850	.99205	NiAl	None	Test: 230hr
Nickel	2	----														NiAl	None	Test: 2270hr
Nickel	3	.9865	Y/Y	.98575	.98780	.98830	.98630	.98440	.98825	.98860	.98810	.98600	.98935	.98660	.99020	NiAl	SS316	Test: 2500hr
Nickel	4	----														NiAl	Ni200	Test: 230hr
Nickel	5	.9885	Y/Y	.99080	.98920	.99170	.99000	.98970	.98840	.99650	.98880	.98980	.98790	.99380	.98860	NiAl	Ni200	Test: 2270hr
Nickel	6	.9875	Y/Y	.98700	.98640	.98820	.98700	.98710	.98700	.98780	.98680	.98730	.98680	.98720	.98655	None	None	Specimen
Nickel	7	.9885	Y/Y	.98920	.98875	.98580	.98770	.98820	.99125	.98580	.98720	.98920	.98980	.98720	.98945	None	None	Test: 2500hr
Nickel	8	.9885	Y/Y	.98585	.98675	.98745	.98625	.98740	.98620	.98780	.98590	.98970	.98585	.98965	.98580	None	SS316	Test: 2500hr
Nickel	9	.9885	Y/Y	.98660	.98580	.98865	.99060	.98780	.98580	.98825	.98885	.98725	.99180	.98620	.98895	None	Ni200	Test: 2500hr
1-24-95, 11:48 AM. Temperature: 296.3 K (74.3 F). Relative Humidity: 43%.																		

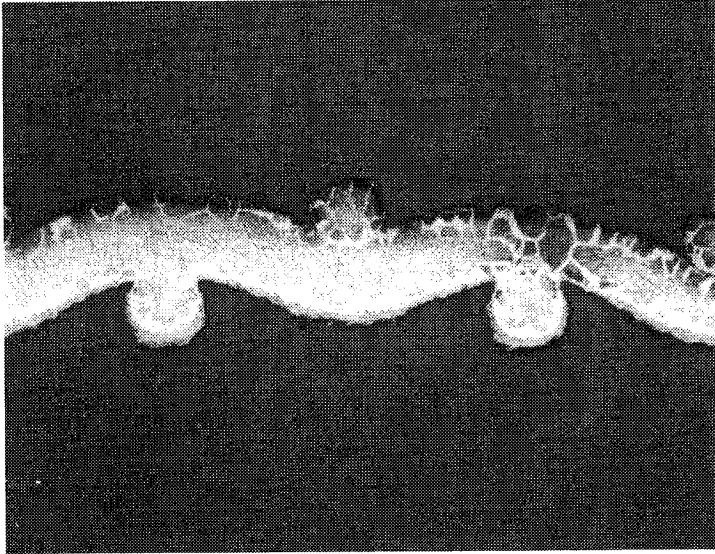
Table B4. Measurement Data for Uncoated and Coated Inconel 718 Sample Tubes After Testing.

Sample Tube		Pin Gauge (Inch)	Zero	At 1.0 Inch Mark (inches)				At 2.0 Inch Mark (inches)				At 3.0 Inch Mark (inches)				Coating Material	Screen Material	Tube Use
Material	Number			A	B	C	D	A	B	C	D	A	B	C	D			
9-12-95, 8:30 AM. Temperature: 296.5 K (74.3 F). Relative Humidity: 60%. JEL, GOB.																		
Inconel	10	.9890	Y/Y	.98840	.98800	.98825	.98740	.98925	.98930	.98910	.98820	.99065	.99000	.99020	.98895	NiAl	None	Test: 8767hr
Inconel	11	.9900	Y/Y	.98800	.98800	.99050	.98805	.99150	.98810	.99250	.98755	.99495	.98760	.99810	.98805	NiAl	SS316	Test: 8767hr
Inconel	12	.9900	Y/Y	.99310	.99520	.99335	.99455	.99360	.99500	.99500	.99590	.99410	.99660	.99570	.99695	NiAl	Ni200	Test: 8767hr
Inconel	13	.9880	Y/Y	.98605	.98655	.98590	.98620	.98605	.98640	.98560	.98640	.98605	.98640	.98605	.98640	NiAl	Ni200	Specimen
Inconel	14	.9890	Y/Y	.98655	.98755	.98815	.98760	.98680	.98815	.98835	.98865	.98840	.98875	.98840	.98880	None	None	Test: 8767hr
Inconel	15	.9880	Y/Y	.99010	.98615	.98680	.98650	.99010	.98645	.98675	.98650	.98895	.98705	.98680	.98670	None	None	Specimen
Inconel	16	.9880	Y/Y	.98820	.98770	.98830	.98815	.99000	.98845	.98790	.98855	.99060	.98875	.98925	.98860	None	SS316	Test: 8767hr
Inconel	17	.9900	Y/Y	.99200	.99300	.99210	.99320	.99345	.99225	.99300	.99300	.99235	.99080	.99510	.99360	None	Ni200	Test: 8767hr
9-12-95, 9:15 AM. Temperature: 298.2 K (77.3 F). Relative Humidity: 60%.																		

Table B5. Measurement Data for Uncoated and Coated Udimet 720L Sample Tubes After Testing.																			
Sample Tube	Material	Number	Pin Gauge (inch)	Zero	At 1.0 Inch Mark (inches)				At 2.0 Inch Mark (inches)				At 3.0 Inch Mark (inches)				Coating Material	Screen Material	Tube Use
					A	B	C	D	A	B	C	D	A	B	C	D			
2-24-95 Uncoated and Coated Ni200 sample tubes placed in Quality Assurance room at Thermacore.																			
2-27-95, 1:11 PM. Temperature: 295.3 K (72.2 F). Relative Humidity: 36%. JEL, GOB.																			
Udimet		18		.9875	.98740	.98740	.98755	.98710	.98745	.98760	.98750	.98735	.98770	.98745	.98790	.98800	NiAl	SS316	Specimen
Udimet		19		.9895	.99360	.99375	.99360	.99350	.99375	.99400	.99375	.99500	.99580	.99575	.99650	.99360	NiAl	None	Test: 1886hr
Udimet		20	--														NiAl	Ni200	Specimen
Weld and braze test specimen																			
Udimet		21		.9895	.98835	.98830	.98910	.98840	.99050	.98825	.99060	.98700	.99060	.98895	.99130	.98805	NiAl	SS316	Test: 1886hr
Udimet		22		.9895	.98880	.99120	.99010	.99300	.98910	.99350	.99340	.99425	.98945	.99750	.99700	.99725	NiAl	Ni200	Test: 1886hr
Udimet		23		.9895	.99060	.99050	.99030	.99020	.99065	.98985	.99070	.99030	.98990	.99100	.99020	.99160	None	None	Test: 1886hr
Udimet		24		.9895	.98865	.99265	.98890	.99010	.98910	.99135	.98980	.99040	.99130	.99120	.99150	.99005	None	SS316	Test: 1886hr
Udimet		25		.9895	.98980	.98940	.99280	.99025	.98960	.99090	.99170	.99190	.99005	.99235	.99150	.99225	None	Ni200	Test: 1886hr
Udimet		26		.9875													None	None	Specimen
2-27-95, 2:05 PM. Temperature: 295.7 K (72.9 F). Relative Humidity: 40%.																			

APPENDIX C

NICKEL 200, INCONEL 718 AND UDIMET 720LI SOLUBILITY CORROSION TEST VEHICLES, PHOTOMICROGRAPHS FOR TESTED SS316 AND NI200 SAMPLE TUBE SCREENS



Task 2

Sample No. 27

Ni200 Test Vehicle.

Sample Tube No. 3

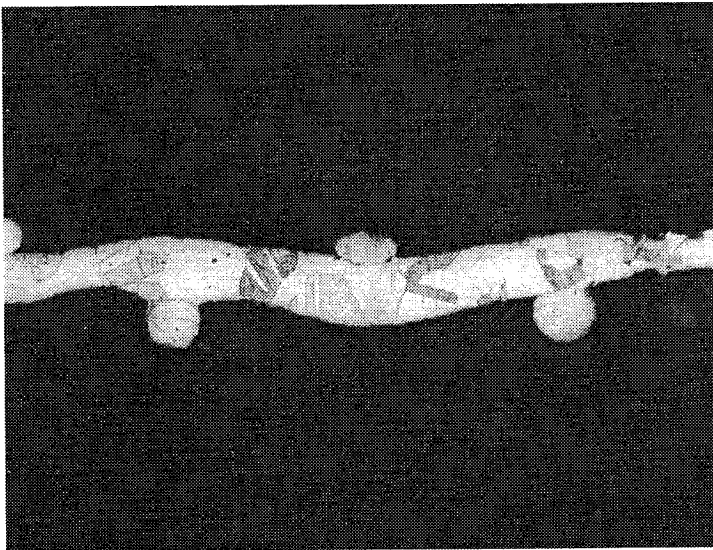
1.0in from end cap.

Uncoated SS316 screen.

Tested 2500 hours at 1073K.

Magnification: 100x

Etched



Task 2

Sample No. 28

Ni200 Test Vehicle.

Sample Tube No. 5

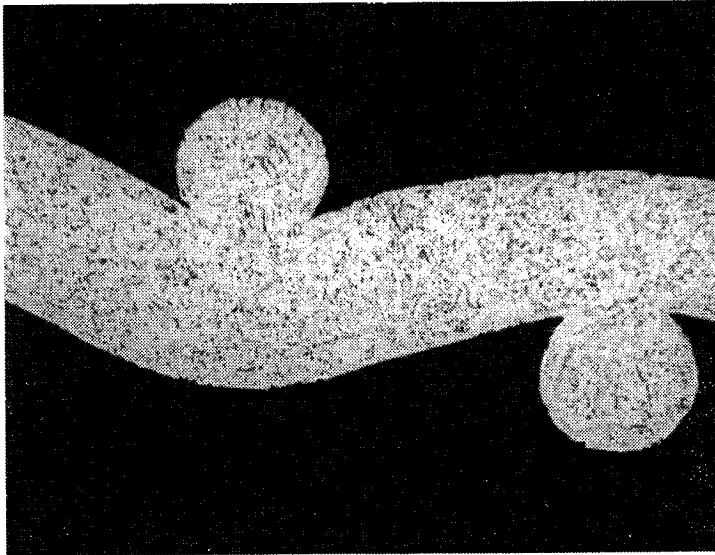
1.0in from end cap.

Uncoated Ni200 screen.

Tested 2270 hours at 1073K.

Magnification: 100x

Etched



Task 2

Sample No. 39

Ud720LI Test Vehicle.

Sample Tube No. 21

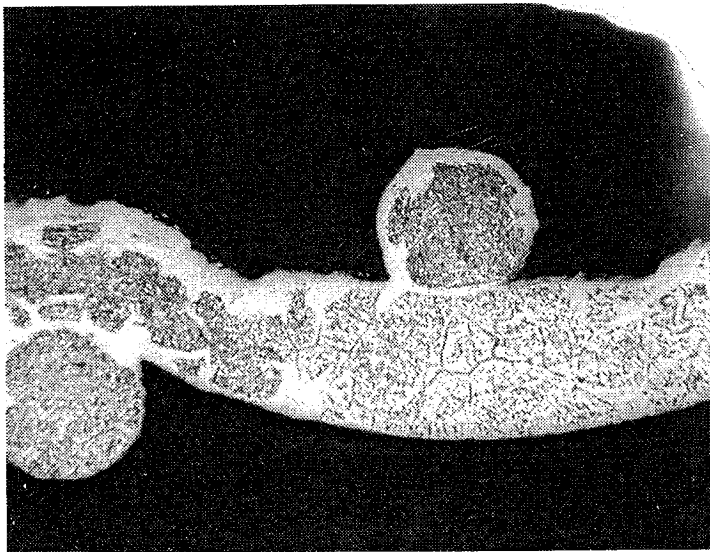
3.0in from end cap.

Uncoated SS316 screen.

Tested 1886 hours at 1073K.

Magnification: 200x

Etched



Task 2

Sample No. 43

Ud720LI Test Vehicle.

Sample Tube No. 24

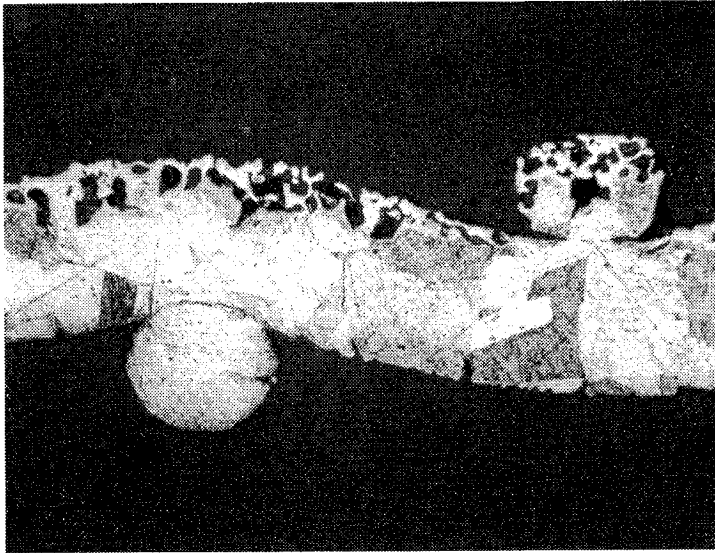
1.0in from end cap.

Uncoated SS316 screen.

Tested 1886 hours at 1073K.

Magnification: 200x

Etched



Task 2

Sample No. 48

Ud720LI Test Vehicle.

Sample Tube No. 25

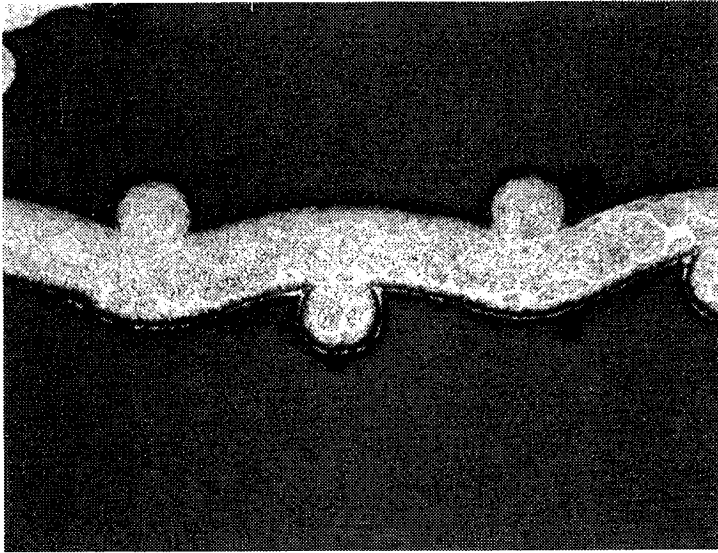
3.0in from end cap.

Uncoated Ni200 screen.

Tested 1886 hours at 1073K.

Magnification: 200x

Etched



Task 2

Sample No. 98

In718 Test Vehicle.

Sample Tube No. 11

1.0in from end cap.

Uncoated SS316 screen.

Tested 8767 hours at 1073K.

Magnification: 100x

Etched

Ni200 screen reduced to dust after 8767 hours.
No Photomicrographs

Task 2

Sample No. N/A

In718 Test Vehicle.

Sample Tube No. N/A

No Location.

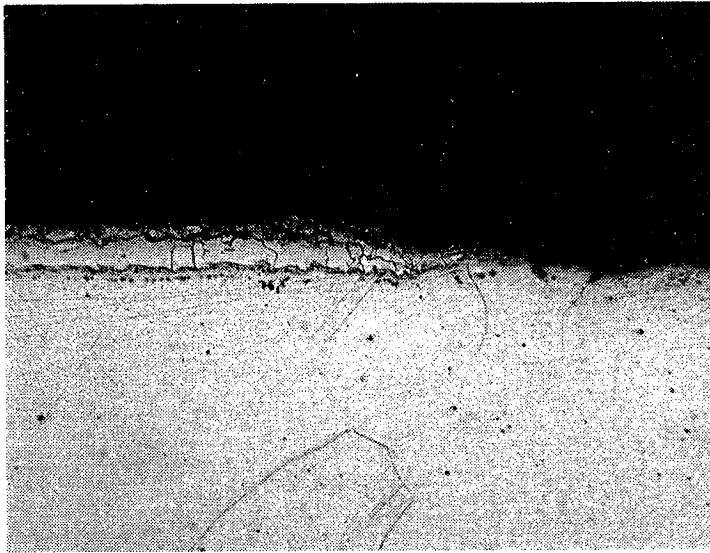
Uncoated Ni200 screen.

Tested 8767 hours at 1073K.

Magnification: N/A

APPENDIX D

NICKEL 200, INCONEL 718 AND UDIMET 720LI SOLUBILITY CORROSION TEST VEHICLES, PHOTOMICROGRAPHS FOR EDGE OF REFERENCE STRIPE SAMPLES



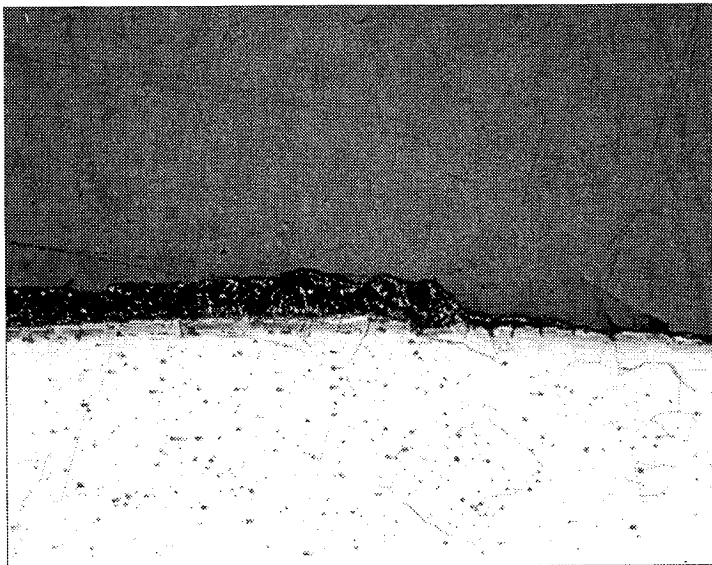
Task 2
Sample No. 3a, edge

Ni200 Test Vehicle.
Sample Tube No. 1
2-step nickel aluminide coating.
Four tungsten reference stripes.
3.0in from end cap.

Coating thickness: 0.00318cm.
Stripe detached from coating.
No reference line.

Tested 230 hours at 1073K.

Magnification: 200x
Etched



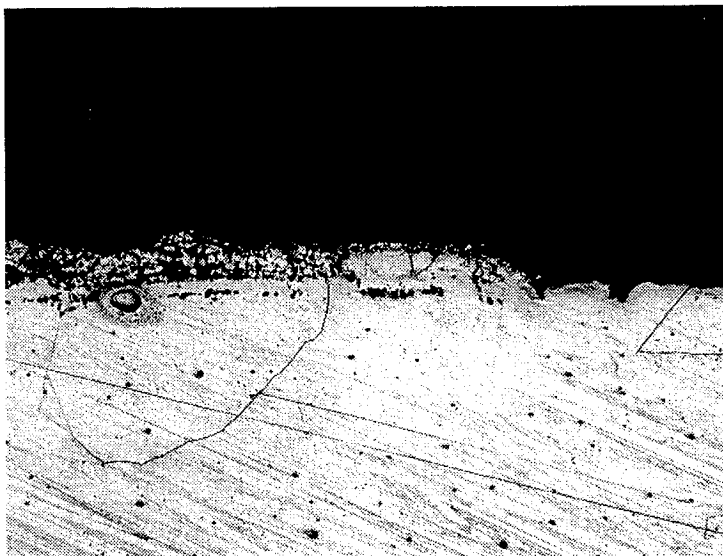
Task 2
Sample No. 4b1, edge

Ni200 Test Vehicle.
Sample Tube No. 2
2-step nickel aluminide coating.
Four tungsten reference stripes.
1.0in from end cap.

Coating thickness: 0.00318cm.
Stripe thickness: 0.00635cm.
Est corrosion rate: 0.0061cm/yr.

Tested 2270 hours at 1073K.

Magnification: 100x
Etched



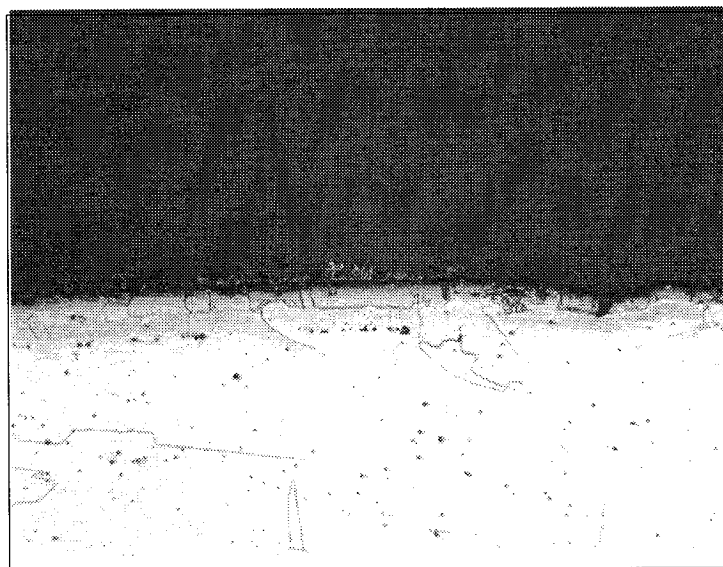
Task 2
Sample No. 5a, edge

Ni200 Test Vehicle.
Sample Tube No. 2
2-step nickel aluminide coating.
Four tungsten reference stripes.
2.0in from end cap.

Coating thickness: 0.00318cm.
Stripe thickness: 0.00635cm.
Est corrosion rate: 0.0061cm/yr.

Tested 2270 hours at 1073K.

Magnification: 100x
Etched



Task 2

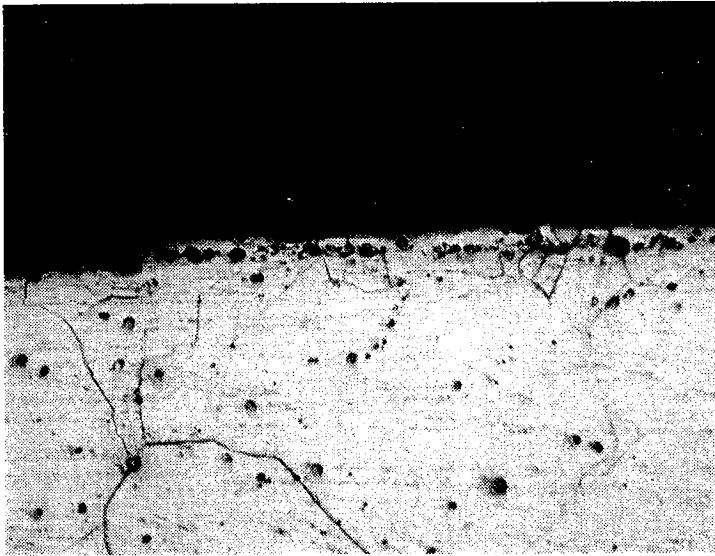
Sample No. 12a, edge

Ni200 Test Vehicle.
Sample Tube No. 5
2-step nickel aluminide coating.
Four tungsten reference stripes.
3.0in from end cap.

Coating thickness: 0.00318cm.
Stripe thickness: 0.00318cm.
Est corrosion rate: 0.0030cm/yr.

Tested 2270 hours at 1073K.

Magnification: 100x
Etched



Task 2

Sample No. 18b, edge

Ni200 Test Vehicle.

Sample Tube No. 7.

Uncoated.

2 tungsten / 2 nickel ref. stripes.

3.0in from end cap.

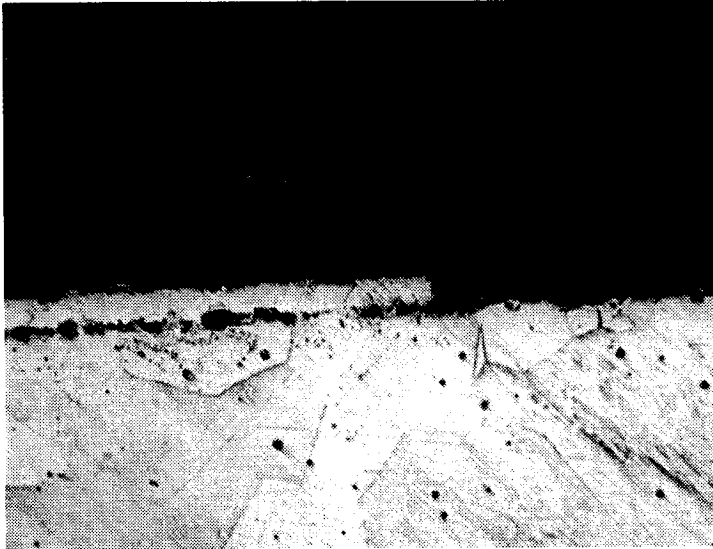
Stripe thickness: 0.00157cm.

Est corrosion rate: 0.0028cm/yr.

Tested 2500 hours at 1073K.

Magnification: 200x

Etched



Task 2

Sample No. 22a, edge

Ni200 Test Vehicle.

Sample Tube No. 9

Uncoated.

2 tungsten / 2 nickel ref. stripes.

1.0in from end cap.

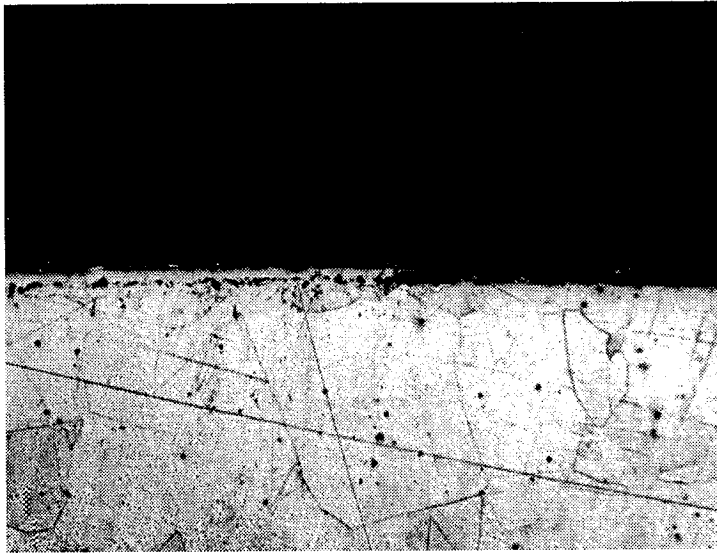
Stripe thickness: 0.00239cm.

Est corrosion rate: 0.0028cm/yr.

Tested 2500 hours at 1073K.

Magnification: 200x

Etched



Task 2

Sample No. 23a, edge

Ni200 Test Vehicle.

Sample Tube No. 9

Uncoated.

2 tungsten / 2 nickel ref. stripes.

2.0in from end cap.

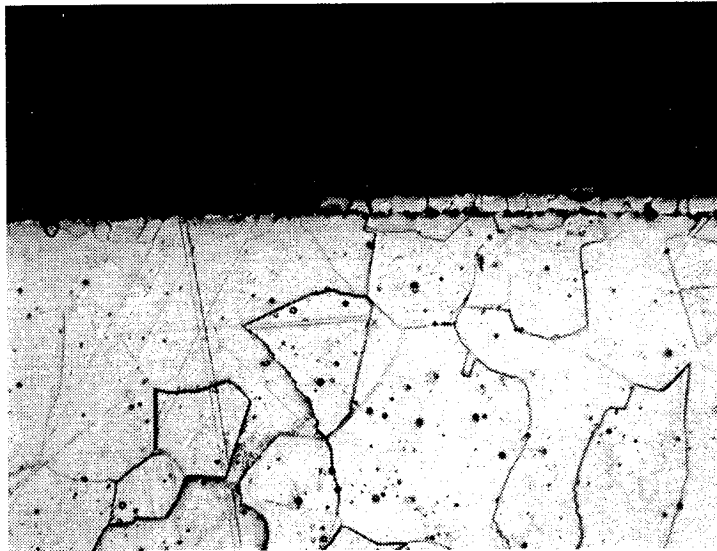
Stripe thickness: 0.00157cm.

Est corrosion rate: 0.0028cm/yr.

Tested 2500 hours at 1073K.

Magnification: 100x

Etched



Task 2

Sample No. 24a, edge

Ni200 Test Vehicle.

Sample Tube No. 9

Uncoated.

2 tungsten / 2 nickel ref. stripes.

3.0in from end cap.

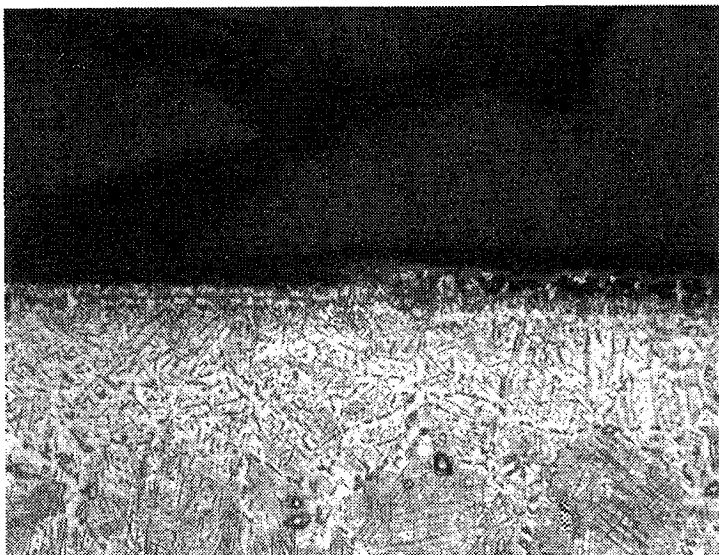
Stripe thickness: 0.00318cm.

Est corrosion rate: 0.0028cm/yr.

Tested 2500 hours at 1073K.

Magnification: 100x

Etched



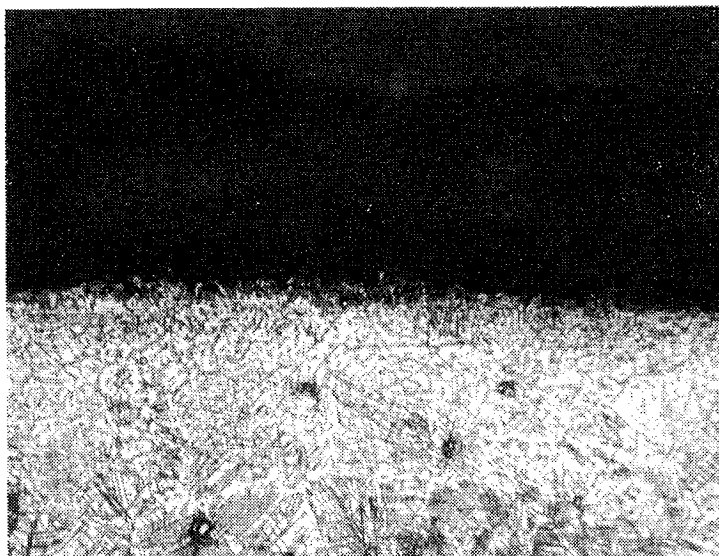
Task 2
Sample No. 73b, edge

In718 Test Vehicle.
Sample Tube No. 10
2-step nickel aluminide coating.
Four tungsten reference stripes.
1.0in from end cap.

Coating thickness: 0.00157cm.
Stripe thickness: 0.00157cm.
No visible signs of corrosion.

Tested 8767 hours at 1073K.

Magnification: 200x
Etched



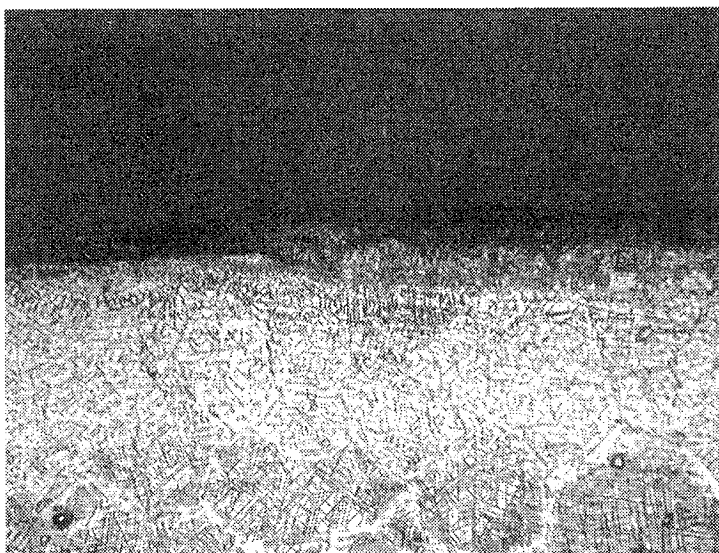
Task 2
Sample No. 74b, edge

In718 Test Vehicle.
Sample Tube No. 10
2-step nickel aluminide coating.
Four tungsten reference stripes.
2.0in from end cap.

Coating thickness: 0.00157cm.
Stripe thickness: 0.00079cm.
Est corrosion rate: 0.00079cm/yr.

Tested 8767 hours at 1073K.

Magnification: 200x
Etched

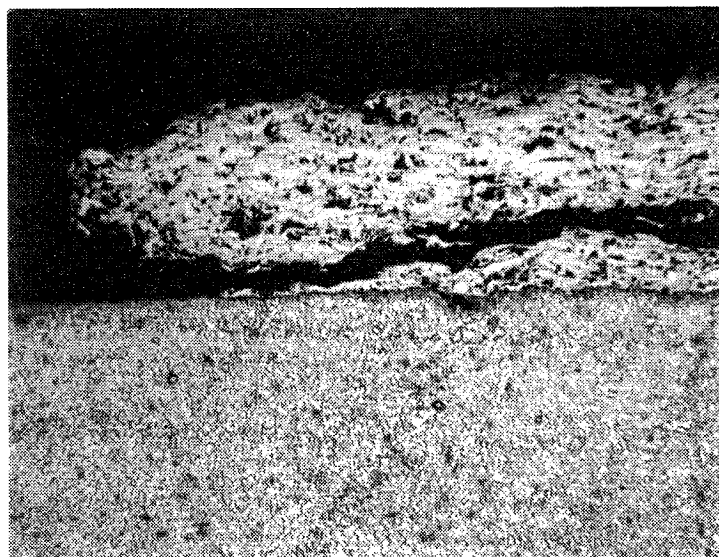


Task 2
Sample No. 77b, edge

In718 Test Vehicle.
Sample Tube No. 11
2-step nickel aluminide coating.
Four tungsten reference stripes.
2.0in from end cap.

Coating thickness: 0.00157cm.
Stripe thickness:
0.00079cm to 0.00157cm.
No visible signs of corrosion.
Tested 8767 hours at 1073K.

Magnification: 200x
Etched



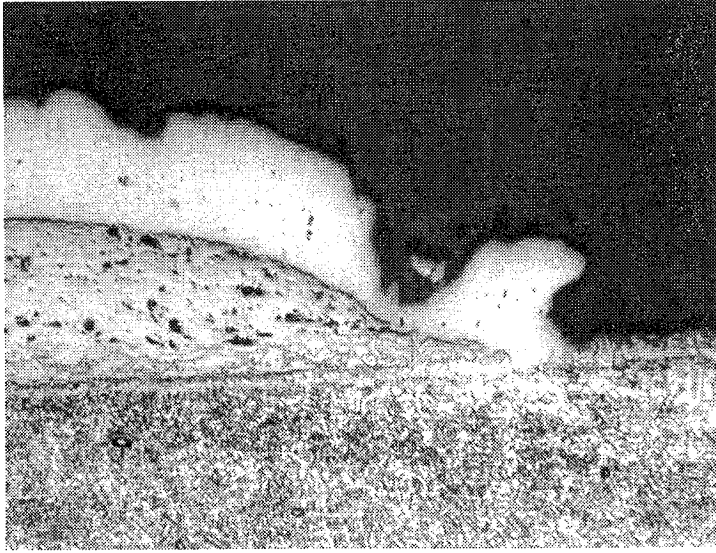
Task 2
Sample No. 87b, edge

In718 Test Vehicle.
Sample Tube No. 14
Uncoated.
Four tungsten reference stripes.
3.0in from end cap.

Stripe was split.
Stripe thickness: 0.01270cm.
No visible signs of corrosion.

Tested 8767 hours at 1073K.

Magnification: 200x
Etched



Task 2

Sample No. 94b, edge

In718 Test Vehicle.

Sample Tube No. 17

Uncoated.

Four tungsten reference stripes.

1.0in from end cap.

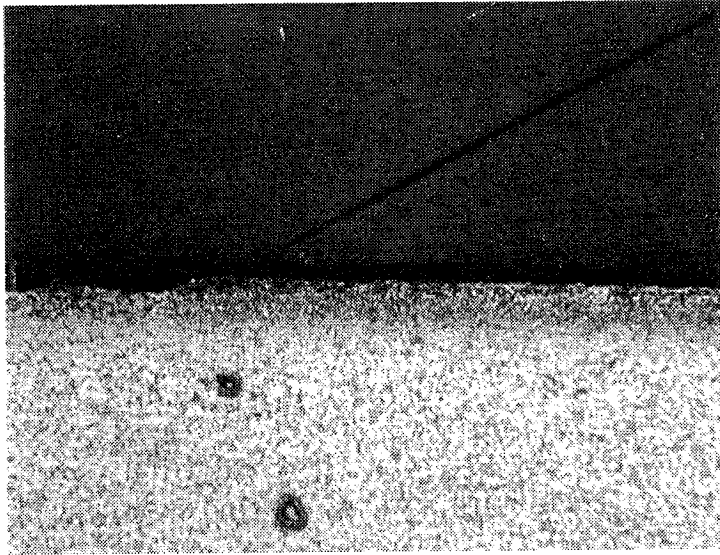
Stripe thickness: 0.01270cm.

No visible signs of corrosion.

Tested 8767 hours at 1073K.

Magnification: 200x

Etched



Task 2
Sample No. 52, edge

Ud720LI Test Vehicle.
Sample Tube No. 19
2-step nickel aluminide coating.
Four tungsten reference stripes.
1.0in from end cap.

Coating thickness:
0.00079cm to 0.00157cm.
Stripe detached from coating.
No reference line.
Tested 1886 hours at 1073K.

Magnification: 200x
Etched



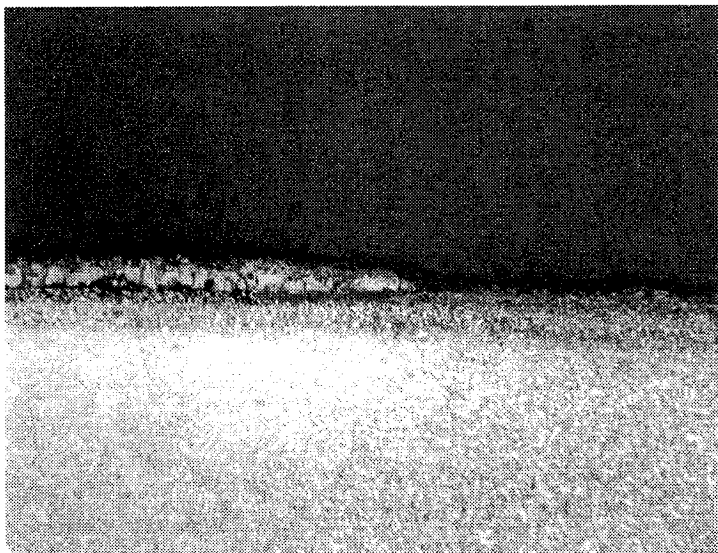
Task 2
Sample No. 56, edge

Ud720LI Test Vehicle.
Sample Tube No. 21
2-step nickel aluminide coating.
Four tungsten reference stripes.
2.0in from end cap.

Coating thickness: 0.00157cm.
Stripe split within thickness.
No visible signs of corrosion.

Tested 1886 hours at 1073K.

Magnification: 200x
Etched



Task 2
Sample No. 58, edge

Ud720LI Test Vehicle.
Sample Tube No. 22
2-step nickel aluminide coating.
Four tungsten reference stripes.
1.0in from end cap.

Coating thickness:
0.00157cm to 0.00239cm.
Stripe split within thickness.
No visible signs of corrosion.
Tested 1886 hours at 1073K.

Magnification: 200x
Etched

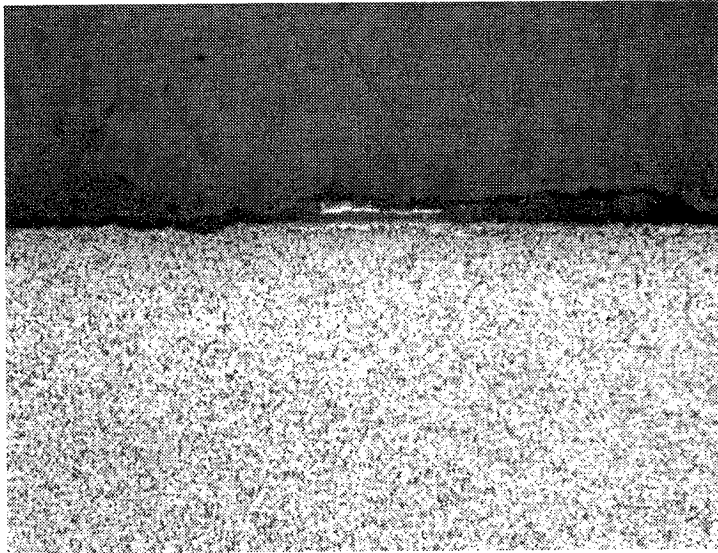


Task 2
Sample No. 60, edge

Ud720LI Test Vehicle.
Sample Tube No. 22
2-step nickel aluminide coating.
Four tungsten reference stripes.
3.0in from end cap.

Coating thickness:
0.00079cm to 0.00239cm.
Stripe thickness:
0.00157cm to 0.00318cm.
No visible signs of corrosion.
Tested 1886 hours at 1073K.

Magnification: 200x
Etched



Task 2

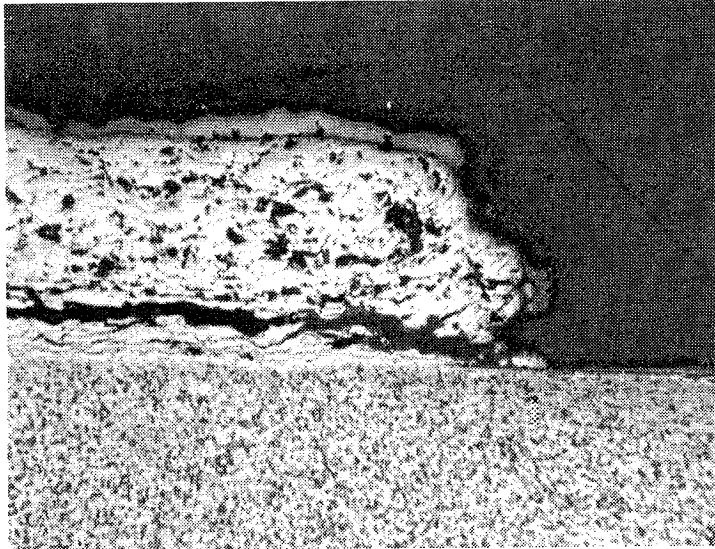
Sample No. 64, edge

Ud720LI Test Vehicle.
Sample Tube No. 24
Uncoated.
Four tungsten reference stripes.
1.0in from end cap.

Stripe split within thickness.
No visible signs of corrosion.

Tested 1886 hours at 1073K.

Magnification: 200x
Etched



Task 2

Sample No. 68, edge

Ud720LI Test Vehicle.
Sample Tube No. 25
Uncoated.
Four tungsten reference stripes.
2.0in from end cap.

Stripe thickness: 0.01422cm

No visible signs of corrosion.

Tested 1886 hours at 1073K.

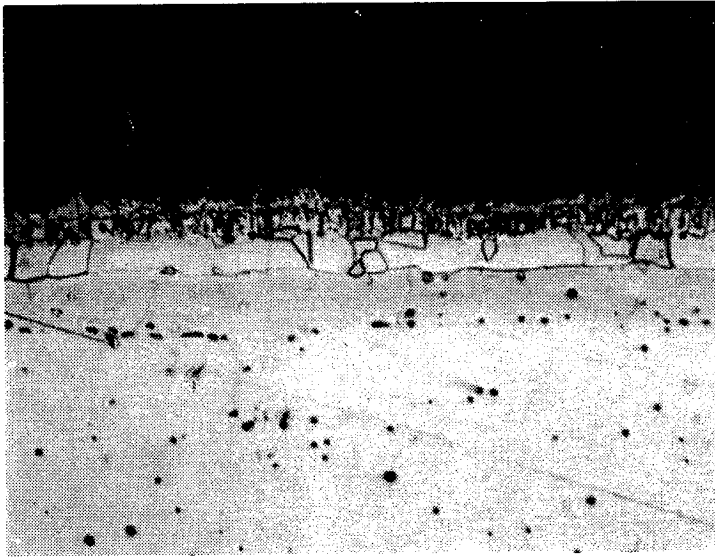
Magnification: 200x
Etched

APPENDIX E

NICKEL 200, INCONEL 718 AND UDIMET 720LI SOLUBILITY CORROSION TEST VEHICLES,

PHOTOMICROGRAPHS FOR CENTER OF REFERENCE STRIPE

AND COATING SAMPLES



Task 2
Sample No. 11a, coating

Ni200 Test Vehicle.
Sample Tube No. 5
2-step nickel aluminide coating.
Four tungsten reference stripes.
2.0in from end cap.

Coating thickness: 0.00635cm
Stripe not shown.
Cannot determine corrosion.

Tested 2270 hours at 1073K.

Magnification: 200x
Etched



Task 2

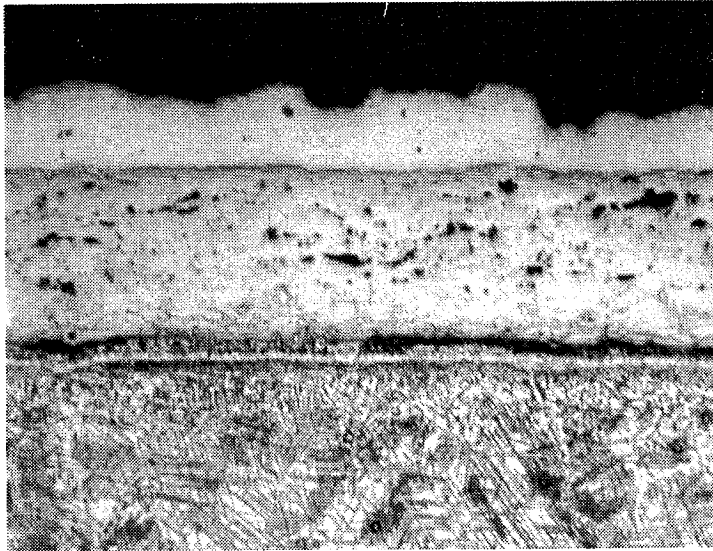
Sample No. 17a, stripe

Ni200 Test Vehicle.
Sample Tube No. 7
Uncoated.
2 tungsten / 2 nickel ref. stripes.
2.0in from end cap.

Tungsten Stripe thickness:
0.00475cm to 0.00635cm.
Cannot determine corrosion.

Tested 2500 hours at 1073K.

Magnification: 100x
Etched



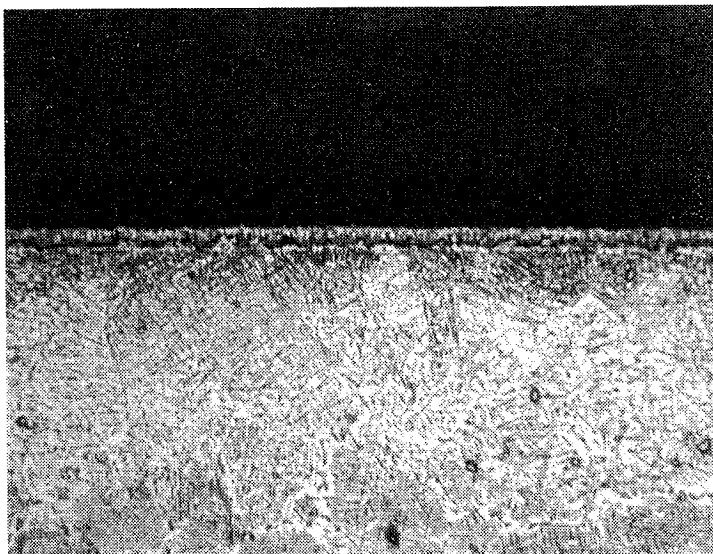
Task 2
Sample No. 79, stripe

In718 Test Vehicle.
Sample Tube No. 12
2-step nickel aluminide coating.
Four tungsten reference stripes.
1.0in from end cap.

Coating thickness: 0.00157cm
Stripe thickness: 0.01270cm
Cannot determine corrosion.

Tested 8767 hours at 1073K.

Magnification: 200x
Etched



Task 2

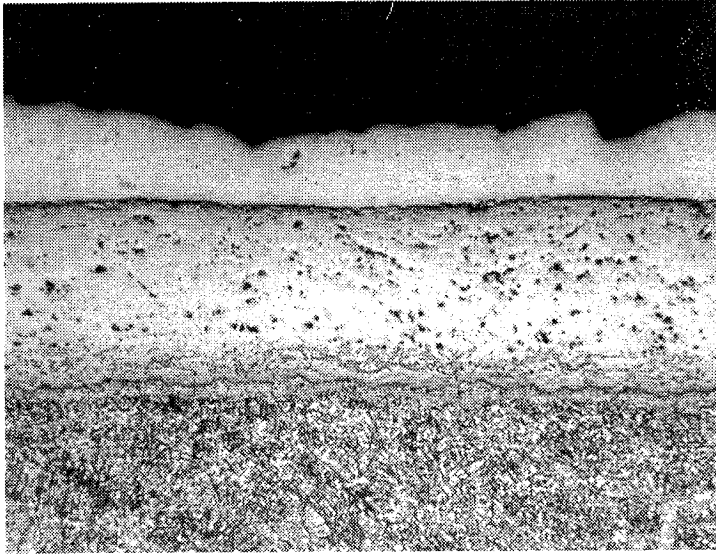
Sample No. 76a, stripe

In718 Test Vehicle.
Sample Tube No. 11
2-step nickel aluminide coating.
Four tungsten reference stripes.
1.0in from end cap.

Coating thickness: 0.00157cm
Stripe thickness: 0.00157cm.
Cannot determine corrosion.

Tested 8767 hours at 1073K.

Magnification: 200x
Etched



Task 2

Sample No. 94a, stripe

In718 Test Vehicle.

Sample Tube No. 17

Uncoated.

Four tungsten reference stripes.

2.0in from end cap.

Stripe thickness: 0.01270cm.

Cannot determine corrosion.

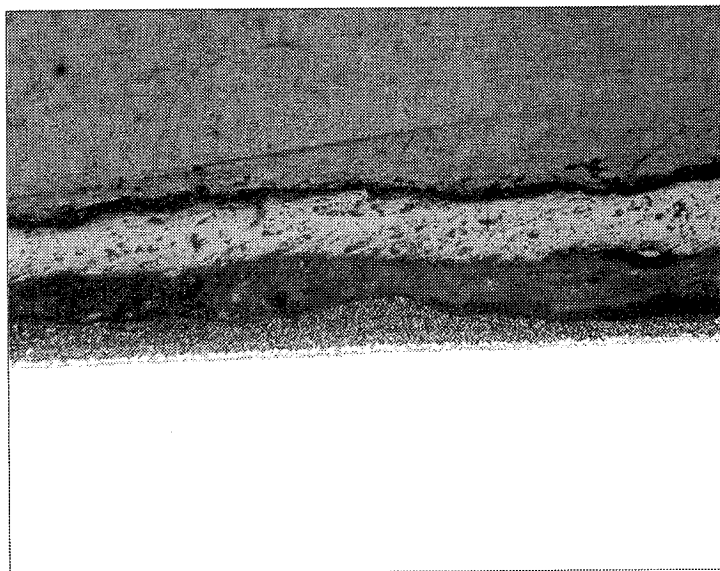
Tested 8767 hours at 1073K.

Magnification: 200x

Etched

APPENDIX F

**PHOTOMICROGRAPHS FOR ALUMINUM OXIDE STRIPES PLASMA-SPRAYED
ONTO NICKEL ALUMINIDE COATED AND UNCOATED INCONEL 718 SUBSTRATES**



Task 2

Unfired

In718

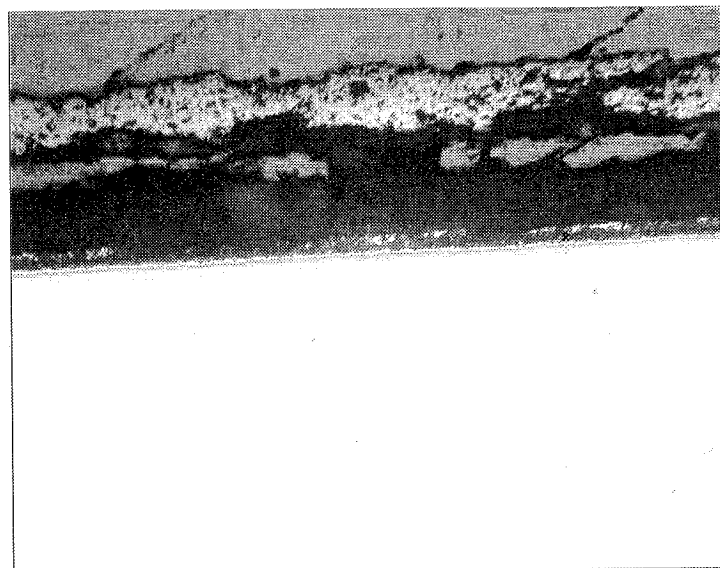
2-step nickel aluminide coating.

Aluminum oxide stripe.

Magnification: 100x

Etched

3
2
1



Task 2

Vacuum fired: 1073K for 8 hr.

In718

2-step nickel aluminide coating.

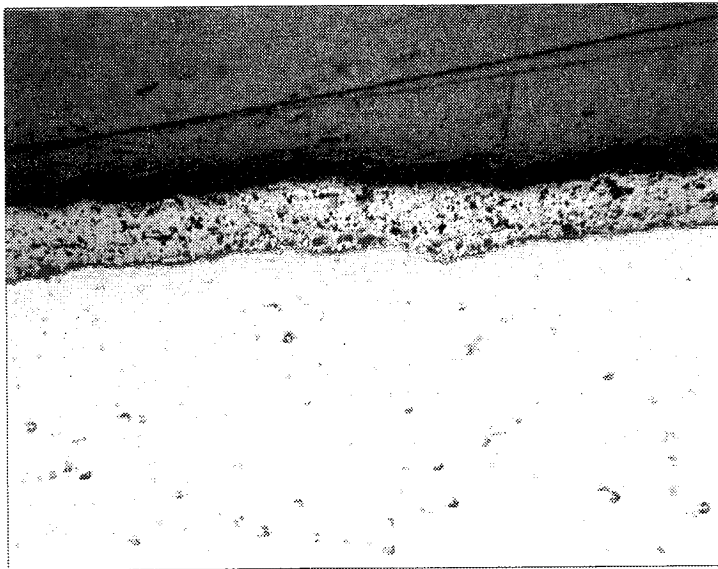
Aluminum oxide stripe.

Magnification: 100x

Etched

3
2
1

- 1 Inconel 718
- 2 Nickel aluminide
- 3 Aluminum oxide



Task 2

Unfired

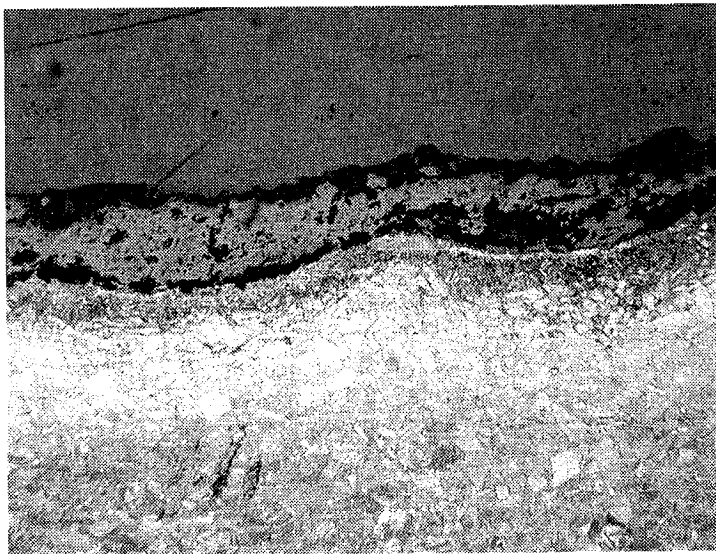
Uncoated In718.

2 Aluminum oxide stripe.

Magnification: 100x

Etched

1



Task 2

Vacuum fired: 1073K for 8hr.

Uncoated In718.

2 Aluminum oxide stripe.

Magnification: 100x

Etched

1

1 Inconel 718
2 Aluminum oxide

APPENDIX G

**COEFFICIENTS OF THERMAL EXPANSION, CTE RATIOS, MELTING POINTS,
AND MELTING POINT RATIOS FOR THE EVALUATED REFERENCE STRIPE MATERIALS**

Coefficients of Thermal Expansion, CTE Ratios, Melting Points, and Melting Point Ratios for the Evaluated Reference Stripe Materials				
Material	Coefficient of Thermal Expansion (μm/m-K)	$\frac{CTE_{Material}}{CTE_{IN718}}$	Melting Point (K)	$\frac{MP_{Material}}{MP_{IN718}}$
Zirconium	5.9	0.35	2125	1.33
Tantalum	6.6	0.40	3287	2.05
Hafnium	6.1	0.37	2500	1.56
Niobium	7.9	0.47	2740	1.71
Ruthenium	9.6	0.57	2523	1.57
Molybdenum	5.7	0.34	2890	1.80
Rhenium	6.6	0.40	3453	2.15
Tungsten	4.6	0.28	3680	2.30
Chromium	9.4	0.56	2130	1.33
Vanadium	10.4	0.62	2175	1.36
Nickel	16.3	0.98	1726	1.08
Iron	14.6	0.87	1809	1.13
Cobalt	14.6	0.87	1768	1.10
Inconel 718	16.7	N/A	1603	N/A

APPENDIX H

CALCULATION OF DIFFUSION OF CHROMIUM INTO NICKEL ALUMINIDE

BASED ON NASA LeRC SUPPLIED DATA

Title: DIFFUSION OF CHROMIUM
IN NICKEL ALUMINIDE

Calculated by: JE Lundermuth Date: 2/16/94

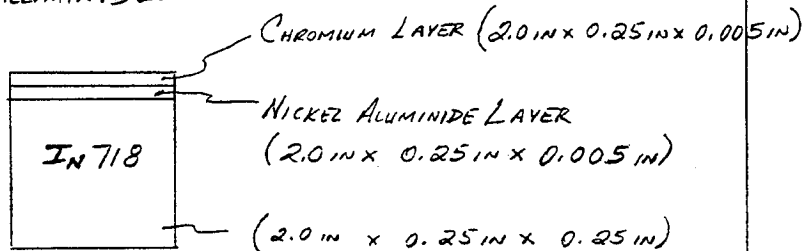
Checked by: _____ Date: _____

Reviewed by: _____ Date: _____

Project: 11-1230 INSOLUBLE COATINGS
PHASE II

Page 1 of

PURPOSE: DETERMINE THE DEPTH TO WHICH CHROMIUM DIFFUSES INTO NICKEL ALUMINIDE.



THE FOLLOWING CASES WILL BE CONSIDERED:

- A) 300 HOURS = 1.08×10^6 SECONDS AT 1273 K
- B) 8760 HOURS = 3.15×10^7 SECONDS AT 1073 K
- C) 8760 HOURS = 3.15×10^7 SECONDS AT 1273 K

ANALYSIS: USING FICK'S SECOND LAW: (MATERIAL SCIENCE FOR ENGINEERS LAWRENCE VAN VLACK, ADDISON - WESLEY PUBLISHING CO 1970, p. 171, EQ 4-3)

$$\frac{\partial C}{\partial t} = \frac{1}{D} \frac{\partial^2 C}{\partial x^2}$$

C = CONCENTRATION

D = DIFFUSION COEFFICIENT

ASSUMPTIONS:

- ① ONE-DIMENSIONAL GRADIENT
- ② D IS INDEPENDENT OF CONCENTRATION
- ③ INFINITE SOURCE OF CHROMIUM FROM PLASMA-SPRAYED STRIPE (I.E., CONSTANT CONCENTRATION OF CHROMIUM AT SURFACE OF NICKEL ALUMINIDE, C_s)
- ④ SEMI-INFINITE NICKEL ALUMINIDE SINK

Title: DIFFUSION OF Cr IN NiAl

 Calculated by: J. L. Lendrum Date: 3/16/94

Checked by: _____ Date: _____

Reviewed by: _____ Date: _____

 Project: 11-1230 - INSOLUBLE COATINGS
PHASE II

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AREA OF DIFFUSION INTO NICKEL ALUMINIDE IS

$$A = (.25 \text{ in})(1.0 \text{ in})(2.54 \text{ cm/in})^2 = 1.61 \text{ cm}^2$$

 DIFFUSION COEFFICIENT, D , FOR CHROMIUM AT 1273 K IS
 APPROXIMATELY $7\text{E}-11 \text{ cm}^2/\text{s}$.

BASED ON DATA SUPPLIED BY NASA LERC.

 ASSUME CONCENTRATION OF CHROMIUM AT SURFACE
 EXPONENTIALLY DECREASES WITH TIME, I.E.,

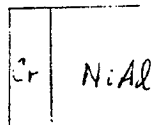
$$(1) \quad C_G(t) = C_{Cr_0} e^{(-\frac{Dt}{A})}$$

 AFTER ONE YEAR: $t = 8760 \text{ HRS} = 3.15\text{E}7 \text{ s}$

$$\frac{Dt}{A} = \frac{(7\text{E}-11 \text{ cm}^2/\text{s})(3.15\text{E}7 \text{ s})}{1.61 \text{ cm}^2} = 1.37\text{E}-3$$

$$C_G = C_{Cr_0} e^{-1.37\text{E}-3} = 0.9986 C_{Cr_0} \approx C_{Cr_0}$$

AS A RESULT ASSUMPTION #3 ON PAGE 1 IS VALID.


 FOR DIFFUSION WITH AN INFINITE SOURCE
 IN A SEMI-INFINITE SINK THE FOLLOWING
 EQUATION CAN BE USED TO DETERMINE
 THE CHROMIUM CONCENTRATION AT A DEPTH
 x INTO THE NICKEL ALUMINIDE (VAN
 VLACK, p.172):

$$(2) \quad \frac{C_s - C_x}{C_s - C_0} = \text{erf} \left(\frac{x}{2\sqrt{Dt}} \right)$$

Title: DIFFUSION OF CHROMIUM IN
NICKEL ALUMINIDE

 Calculated by: JC Landonuth Date: 3/16/84

Checked by: _____ Date: _____

Reviewed by: _____ Date: _____

 Project: 1230 - INSOLUBLE COATINGS Ø II

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$C_0 \equiv$ ORIGINAL CONCENTRATION OF CHROMIUM IN
NICKEL ALUMINIDE

$$C_0 = 0$$

$C_s \equiv$ CONCENTRATION OF CHROMIUM AT SURFACE OF
NICKEL ALUMINIDE

$C_x \equiv$ CONCENTRATION OF CHROMIUM AT A DEPTH x IN
NICKEL ALUMINIDE AT TIME t

$\text{erf}(\) \equiv$ ERROR FUNCTION (SEE TABLE
OF VALUES, P. 6; FROM VAN VLACK, P. 172,
TABLE 9-2)

$$(3) \quad \frac{C_s - C_x}{C_s} = \text{erf} \left(\frac{x}{2\sqrt{Dt}} \right)$$

CASE A) $t = 300 \text{ HRS} = 1.08 \times 10^6 \text{ SECONDS}$
 $T = 1273 \text{ K} \Rightarrow D = 7 \times 10^{-11} \text{ cm}^2/\text{s}$

$$a = 2\sqrt{Dt} = 2\sqrt{(7 \times 10^{-11} \text{ cm}^2/\text{s})(1.08 \times 10^6 \text{ s})} = 1.74 \times 10^{-2} \text{ cm}$$

$x(\text{IN})$	$x(\text{CM})$	$b = \frac{x}{a}$	$\text{erf } b$	C_x
.0008	.002	.11	.12	.88 C_s
.0032	.008	.46	.48	.52 C_s
.0056	.014	.80	.74	.26 C_s
.0088	.022	1.26	.92	.08 C_s

Title: DIFFUSION OF Cr IN NiAl

 Calculated by: JE Lendemann

 Date: 3/16/94

Checked by: _____

Date: _____

Reviewed by: _____

Date: _____

 Project: 11-1236 INSOLUBLE COATINGS
PHASE II

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AS A RESULT, AT 1273 K FOR 300 HOURS THE CHROMIUM WILL DIFFUSE TO A DEPTH OF APPROXIMATELY 0.009 IN. IN NICKEL ALUMINIDE.

CASE B) $t = 8760 \text{ HRS} = 3.15 \times 10^7 \text{ s}$
 $T = 1073 \text{ K} \Rightarrow D = 1 \times 10^{-12} \text{ cm}^2/\text{s}$
 $a = 2\sqrt{Dt} = 2\sqrt{(1 \times 10^{-12})(3.15 \times 10^7)} = 1.12 \times 10^{-2} \text{ cm}$

$x(\text{in})$	$x(\text{cm})$	$b = \frac{x}{a}$	$\text{erf } b$	C_x
.0008	.002	.18	.20	.80 C_s
.0032	.008	.71	.68	.32 C_s
.0056	.014	1.25	.92	.08 C_s

CASE C) $t = 8760 \text{ HRS} = 3.15 \times 10^7 \text{ s}$
 $T = 1273 \text{ K} \Rightarrow D = 7 \times 10^{-11} \text{ cm}^2/\text{s}$
 $a = 9.39 \times 10^{-2} \text{ cm}$

$x(\text{in})$	$x(\text{cm})$	$b = \frac{x}{a}$	$\text{erf } b$	C_x
.008	.020	.21	.23	.77 C_s
.024	.061	.64	.63	.37 C_s
.048	.122	1.30	.93	.07 C_s

A PREAD SHEET FOR CASES A, B, AND C AND A PLOT OF CONCENTRATION VS. DEPTH ARE SHOWN ON PAGES 6 AND 7

11-1230 Insoluble Coatings Phase II

3/30/94

J.E. Lindemuth

CASE A - 300 hours, 1273 K

D	t	$a = 2 \cdot (D \cdot t)^{1/2}$	x	x	$b = (x/a)$	erf(b)	Cx/Cs
(cm ² /s)	(s)	(cm)	(in)	(cm)	()	()	()
7.00E-11	1.08E+06	1.74E-02	0.0000	0.000	0.00	0.00	1.00
7.00E-11	1.08E+06	1.74E-02	0.0008	0.002	0.11	0.13	0.87
7.00E-11	1.08E+06	1.74E-02	0.0016	0.004	0.23	0.25	0.75
7.00E-11	1.08E+06	1.74E-02	0.0024	0.006	0.34	0.37	0.63
7.00E-11	1.08E+06	1.74E-02	0.0032	0.008	0.45	0.48	0.52
7.00E-11	1.08E+06	1.74E-02	0.0040	0.010	0.56	0.57	0.43
7.00E-11	1.08E+06	1.74E-02	0.0048	0.012	0.68	0.66	0.34
7.00E-11	1.08E+06	1.74E-02	0.0056	0.014	0.79	0.74	0.26
7.00E-11	1.08E+06	1.74E-02	0.0064	0.016	0.90	0.80	0.20
7.00E-11	1.08E+06	1.74E-02	0.0072	0.018	1.01	0.85	0.15
7.00E-11	1.08E+06	1.74E-02	0.0080	0.020	1.13	0.89	0.11
7.00E-11	1.08E+06	1.74E-02	0.0088	0.022	1.24	0.92	0.08

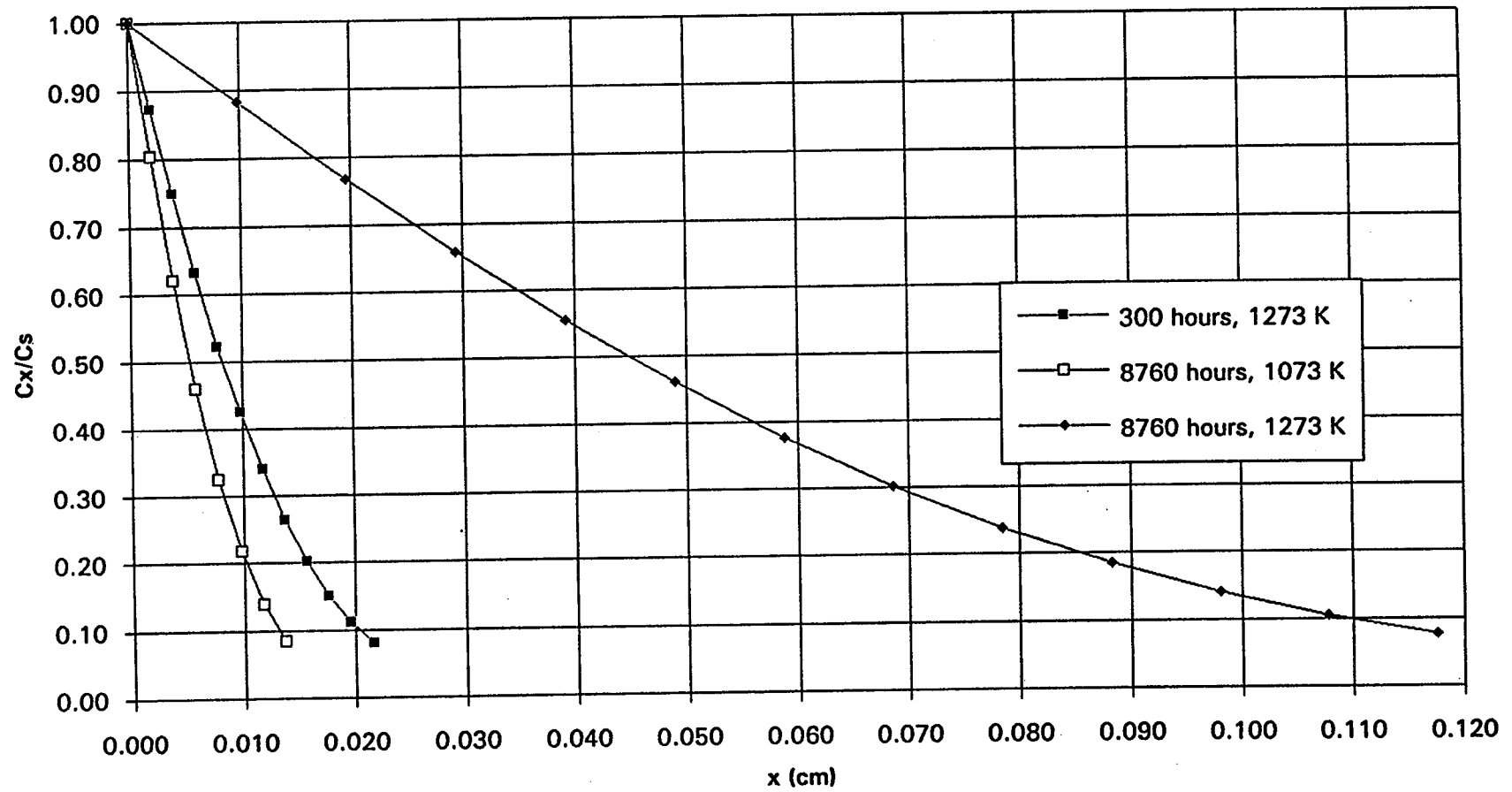
CASE B - 8760 hours, 1073 K

D	t	$a = 2 \cdot (D \cdot t)^{1/2}$	x	x	$b = (x/a)$	erf(b)	Cx/Cs
(cm ² /s)	(s)	(cm)	(in)	(cm)	()	()	()
1.00E-12	3.15E+07	1.12E-02	0.0000	0.000	0.00	0.00	1.00
1.00E-12	3.15E+07	1.12E-02	0.0008	0.002	0.17	0.20	0.80
1.00E-12	3.15E+07	1.12E-02	0.0016	0.004	0.35	0.38	0.62
1.00E-12	3.15E+07	1.12E-02	0.0024	0.006	0.52	0.54	0.46
1.00E-12	3.15E+07	1.12E-02	0.0032	0.008	0.70	0.68	0.32
1.00E-12	3.15E+07	1.12E-02	0.0040	0.010	0.87	0.78	0.22
1.00E-12	3.15E+07	1.12E-02	0.0048	0.012	1.05	0.86	0.14
1.00E-12	3.15E+07	1.12E-02	0.0056	0.014	1.22	0.92	0.08

CASE C - 8760 hours, 1273 K

D	t	$a = 2 \cdot (D \cdot t)^{1/2}$	x	x	$b = (x/a)$	erf(b)	Cx/Cs
(cm ² /s)	(s)	(cm)	(in)	(cm)	()	()	()
7.00E-11	3.15E+07	9.39E-02	0.000	0.000	0.00	0.00	1.00
7.00E-11	3.15E+07	9.39E-02	0.004	0.010	0.10	0.12	0.88
7.00E-11	3.15E+07	9.39E-02	0.008	0.020	0.21	0.23	0.77
7.00E-11	3.15E+07	9.39E-02	0.012	0.029	0.31	0.34	0.66
7.00E-11	3.15E+07	9.39E-02	0.016	0.039	0.42	0.45	0.55
7.00E-11	3.15E+07	9.39E-02	0.020	0.049	0.52	0.54	0.46
7.00E-11	3.15E+07	9.39E-02	0.024	0.059	0.63	0.62	0.38
7.00E-11	3.15E+07	9.39E-02	0.028	0.069	0.73	0.70	0.30
7.00E-11	3.15E+07	9.39E-02	0.032	0.078	0.83	0.76	0.24
7.00E-11	3.15E+07	9.39E-02	0.036	0.088	0.94	0.82	0.18
7.00E-11	3.15E+07	9.39E-02	0.040	0.098	1.04	0.86	0.14
7.00E-11	3.15E+07	9.39E-02	0.044	0.108	1.15	0.90	0.10
7.00E-11	3.15E+07	9.39E-02	0.048	0.118	1.25	0.92	0.08

Diffusion of Chromium in Nickel Aluminide as a Function of Time and Depth.



Title: DIFFUSION OF Cr IN NiAlCalculated by: J. Lindemann Date: 3/16/94

Checked by: _____ Date: _____

Reviewed by: _____ Date: _____

Project: 11-1230 INSOLUBLE COATINGS
PHASE IIPage 8 of _____

CONCLUSIONS:

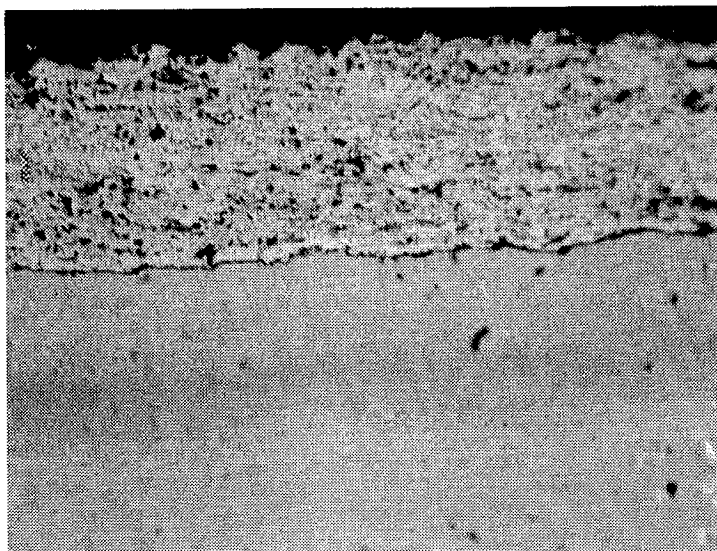
CASE A) $t = 300 \text{ HRS}$
 $T = 1273 \text{ K}$
DEPTH OF CHROMIUM DIFFUSION: 0.009 IN

CASE B) $t = 8760 \text{ HRS}$
 $T = 1073 \text{ K}$
DEPTH: 0.006 IN

CASE C) $t = 8760 \text{ HRS}$
 $T = 1273 \text{ K}$
DEPTH: 0.050 IN

APPENDIX I

PHOTOMICROGRAPHS FOR THE 200 HOUR AND 400 HOUR DIFFUSION TESTS



Task 2

In718

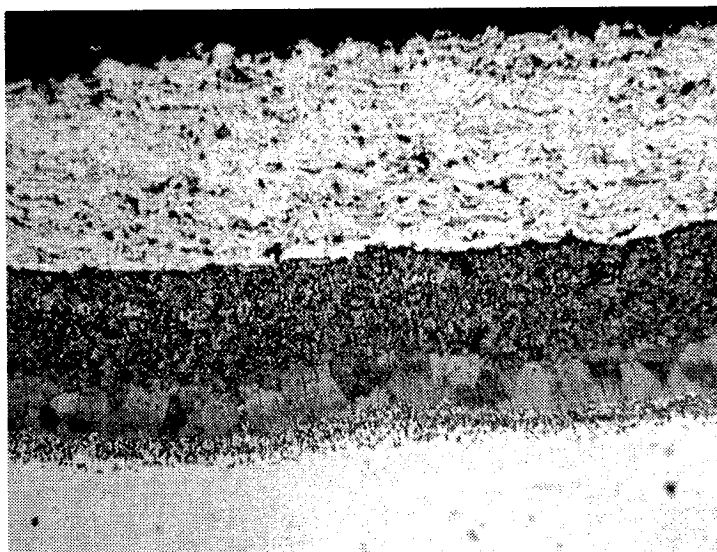
Nickel aluminide coated

Middle of chromium stripe.

1000°C for 207 hours.

Magnification: 200x

Unetched



Task 2

In718

Nickel aluminide coated

Middle of chromium stripe.

1000°C for 207 hours.

Magnification: 200x

Etched

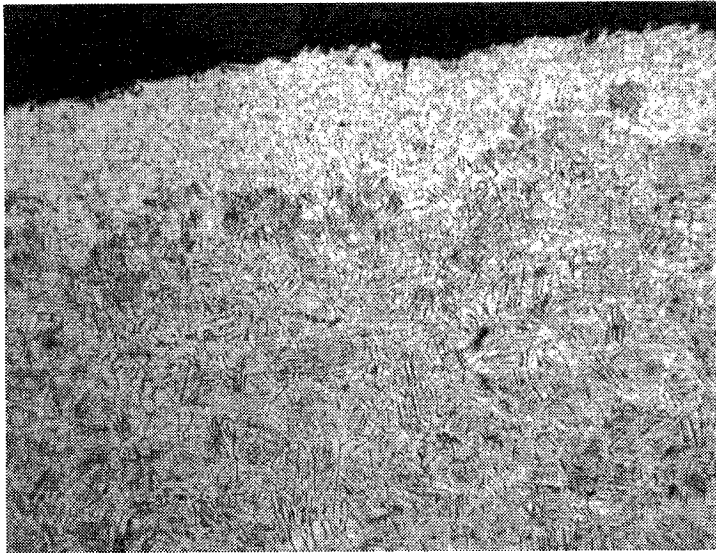
Nickel aluminide

HCl CH₃OH CuCl₂

Chromium

NaOH K₃Fe(CN)₆

- 1 Chromium
- 2 Nickel aluminide
- 3 Transition from nickel aluminide to Inconel 718
- 4 Inconel 718



Task 2

In718

Uncoated

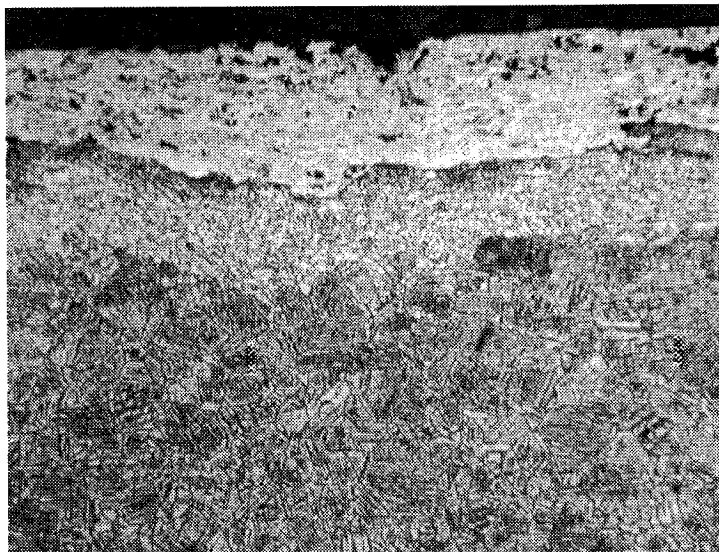
End of sample away from chromium stripe.

1000°C for 207 hours.

Magnification: 200x

Etched

HNO_3 Hcl $\text{CH}_3\text{CO}_2\text{H}$ CuCl_2



Task 2

In718

Uncoated

Middle of chromium stripe.

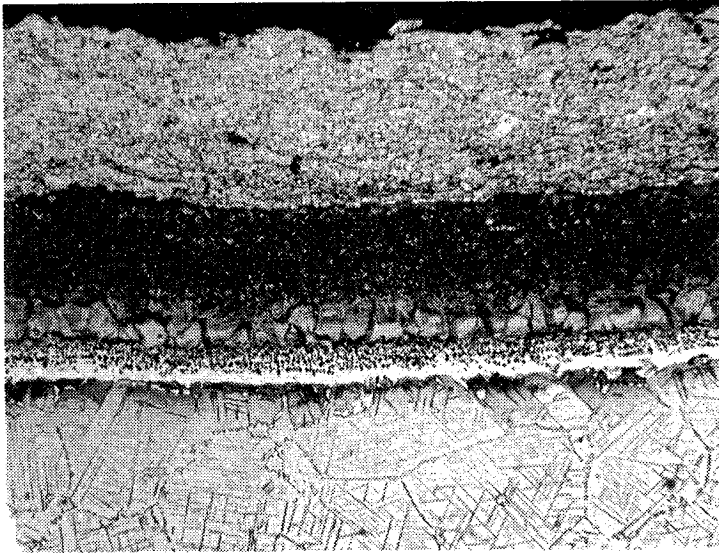
1000°C for 207 hours.

Magnification: 200x

Etched

HNO_3 Hcl $\text{CH}_3\text{CO}_2\text{H}$ CuCl_2

1 Chromium
2 Inconel 718



Task 2

In718

Nickel aluminide coating

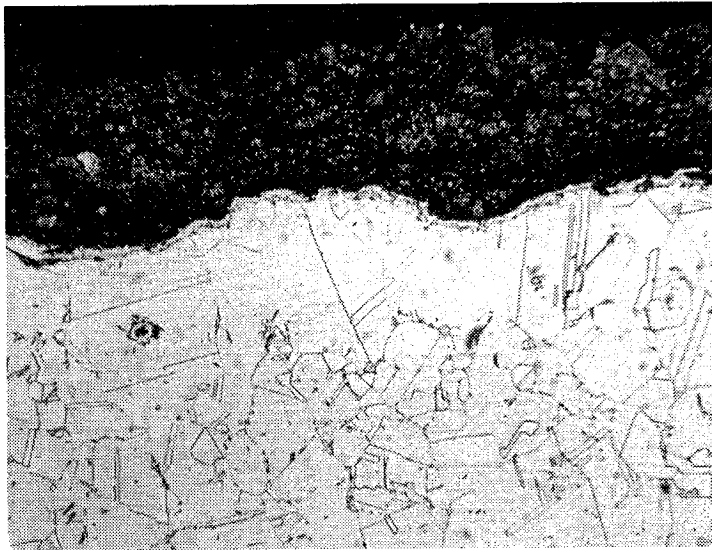
Middle of tungsten stripe.

1000°C for 200 hours.

Magnification: 200x

Etched

HNO_3 HCl $\text{CH}_3\text{CO}_2\text{H}$ CuCl_2



Task 2

In718

Uncoated

Middle of tungsten stripe.

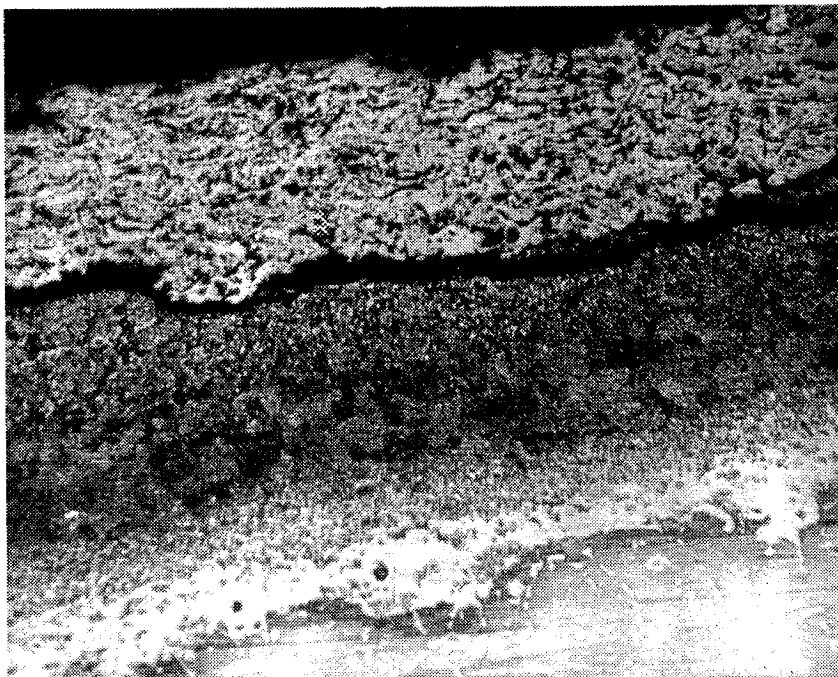
1000°C for 200 hours.

Magnification: 200x

Etched

HNO_3 HCl $\text{CH}_3\text{CO}_2\text{H}$ CuCl_2

- 1 Tungsten
- 2 Nickel aluminide
- 3 Transition from nickel aluminide to Inconel 718
- 4 Inconel 718



Task 2

Sample No. 1

In718: 2-step nickel aluminide coating.

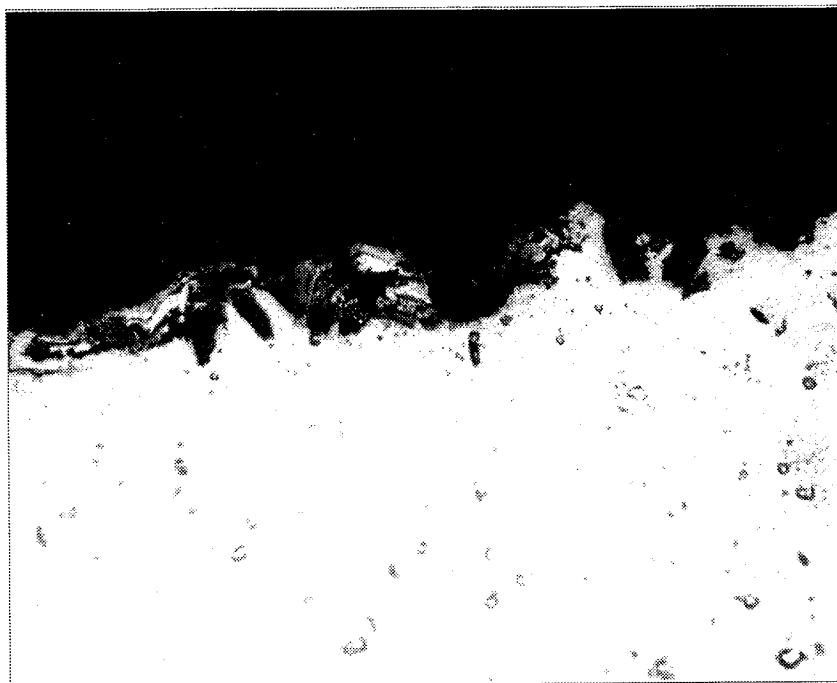
Middle of chromium stripe.

Stripe disbonded during preparation.

1073K for 400 hours.

Magnification: 250x

Etched



Task 2

Sample No. 2

Uncoated In718.

Middle of chromium stripe.

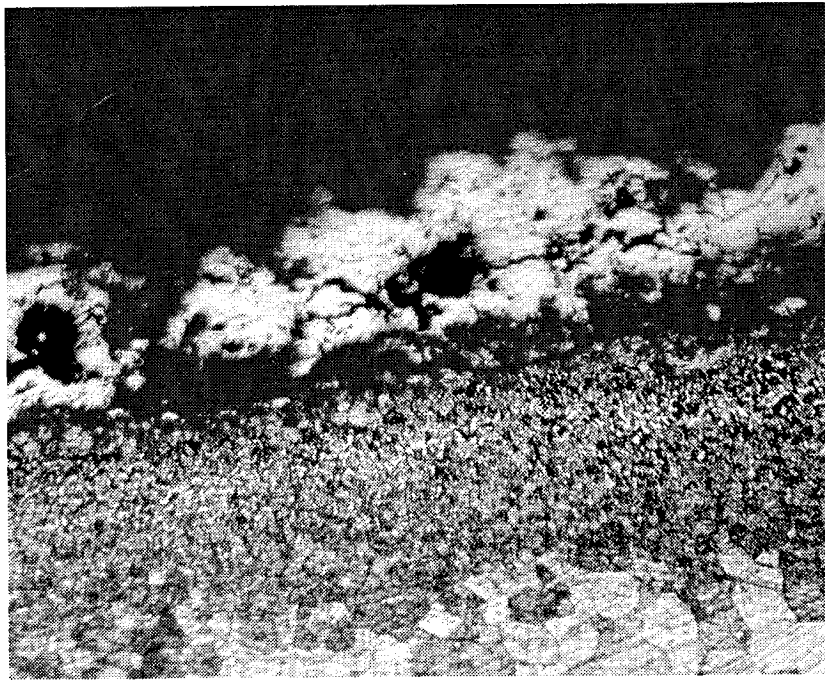
Stripe disbonded during preparation.

1073K for 400 hours.

Magnification: 250x

Etched

- 1 Chromium
- 2 Gap
- 3 Nickel aluminide
- 4 Inconel 718



Task 2

Sample No. 3

1 In718: 2-step nickel aluminide coating.

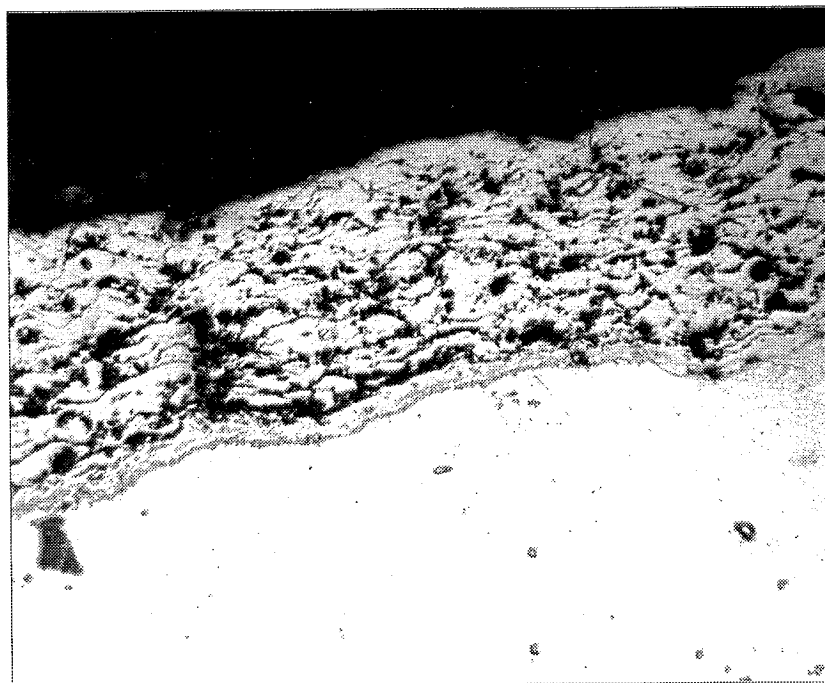
Middle of tungsten stripe.

1073K for 400 hours.

2 Magnification: 250x

Etched

3



Task 2

Sample No. 4

1 Uncoated In718.

Middle of tungsten stripe.

1073K for 400 hours.

3 Magnification: 250x

Etched

3

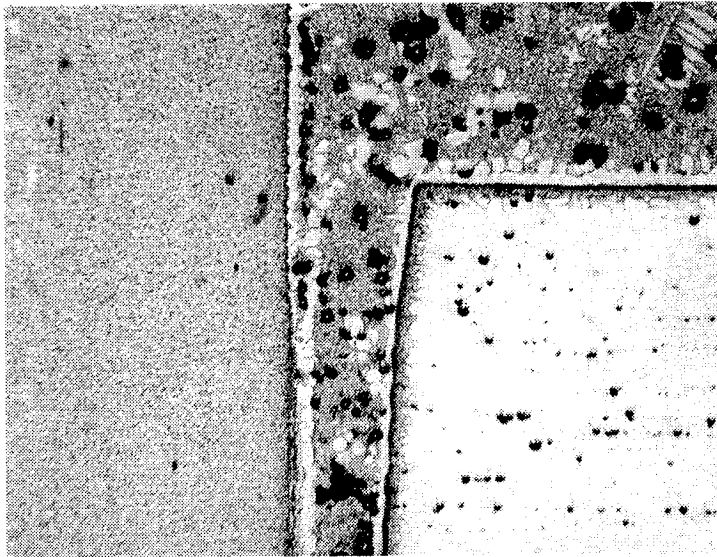
- 1 Tungsten
- 2 Nickel aluminide
- 3 Inconel 718

APPENDIX J

PROCEDURE AND PHOTOMICROGRAPHS FOR THE UDIMET 720LI TO

INCONEL 718 SAMPLE BRAZE EXPERIMENTS

1. Acquire Nicrobraz LM from Thermacore stock or Wall Colmonoy.
2. Machine four Ud720LI tubes and four In718 bases.
3. Nickel plate four Ud720LI tubes using electrolytic nickel plating bath. Plating thickness: 0.0013cm.
4. Nickel plate two In718 bases using electrolytic nickel plating bath. Plating thickness: 0.0013cm.
5. Assemble two nickel plated Ud720LI / unplated In718 braze sample test setups. These samples are Assemblies 1 and 2.
6. Using Nicrobraz LM, braze Assemblies 1 and 2 in vacuum using the following furnace schedule:
 - A. Ramp to 1123K. Soak at 1123K for 15 minutes.
 - B. Ramp to 1173K. Soak at 1173K for 15 minutes.
 - C. Ramp to 1313K. Soak at 1313K for 15 minutes.
 - D. Furnace cool to 313K. Remove assemblies.
7. Helium leak check Assemblies 1 and 2.
8. Section, inspect and photomicrograph Assembly 1.
9. Vacuum fire Assembly 2 at 1373K for four hours.
10. Helium leak check Assembly 2.
11. Section, inspect and photomicrograph Assembly 2.
12. Assemble two nickel plated Ud720LI / nickel plated In718 braze sample test setups. These samples are Assemblies 3 and 4.
13. Using Nicrobraz LM, braze Assemblies 3 and 4 in vacuum using the following furnace schedule:
 - A. Ramp to 1123K. Soak at 1123K for 15 minutes.
 - B. Ramp to 1173K. Soak at 1173K for 15 minutes.
 - C. Ramp to 1313K. Soak at 1313K for 15 minutes.
 - D. Furnace cool to 313K. Remove assemblies.
14. Helium leak check Assemblies 3 and 4.
15. Section, inspect and photomicrograph Assembly 3.
16. Vacuum fire Assembly 4 at 1373K for four hours.
17. Helium leak check Assembly 4.
18. Section, inspect and photomicrograph Assembly 4.



1

2

Task 2

Unfired

Nickel plated Ud720LI tube.

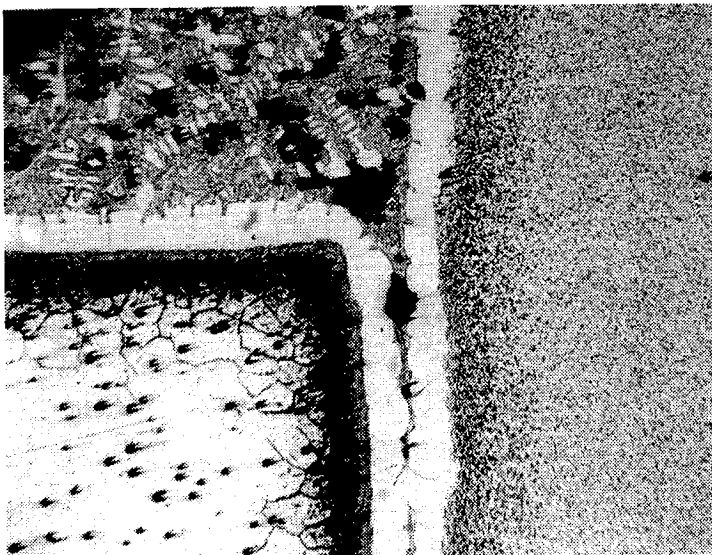
Unplated In718 plate.

Sample No. 1

Brazed using Microbrazz LM.

Magnification: 50x

Etched



2

1

Task 2

Vacuum fired at 1373K for 4 hr.

Nickel plated Ud720LI tube.

Unplated In718 plate.

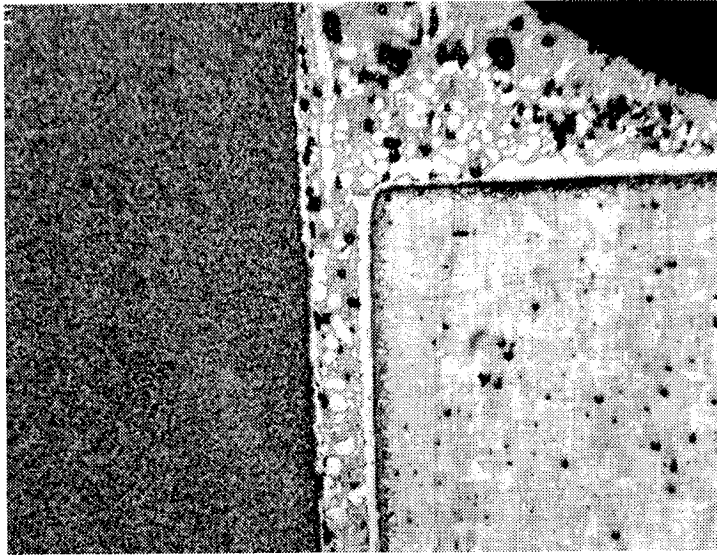
Sample No. 2

Brazed using Microbrazz LM.

Magnification: 50x

Etched

1 Udimet 720LI
2 Inconel 718



1

2

Task 2

Unfired

Nickel plated Ud720LI tube.

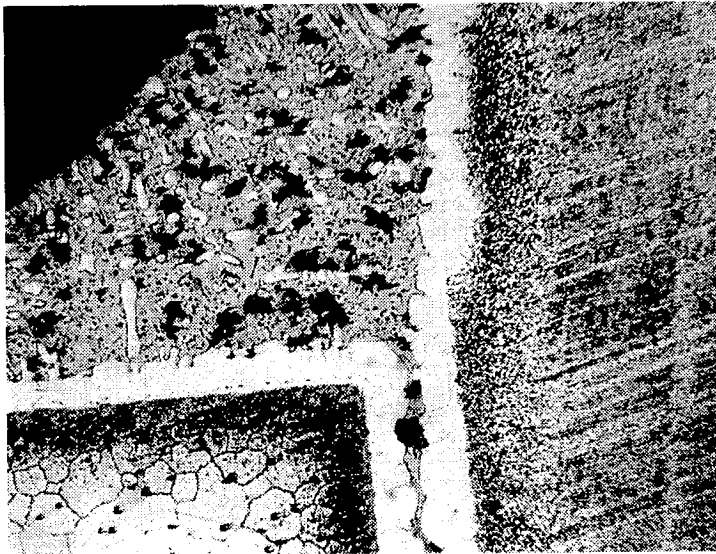
Nickel plated In718 plate.

Sample No. 3

Brazed using Microbrazing LM.

Magnification: 50x

Etched



2

1

Task 2

Vacuum fired at 1373K for 4 hr.

Nickel plated Ud720LI tube.

Nickel plated In718 plate.

Sample No. 4

Brazed using Microbrazing LM.

Magnification: 50x

Etched

1 Udimet 720LI
2 Inconel 718

APPENDIX K

PROCEDURE AND PHOTOMICROGRAPHS FOR THE UDIMET 720LI TO

INCONEL 718 SAMPLE WELD EXPERIMENTS

1. Machine four Ud720LI tubes and four In718 plates.
2. Pickle Ud720LI and In718 parts in a solution of 8 parts HNO_3 to 1.25 parts, 40% HF for 20 minutes to remove oxides. Rinse with deionized water. Dry parts.
3. Assemble four Ud720LI / In718 weld sample test setups.
4. Stake Ud720LI tubes into In718 bases.
5. Stamp each base with a unique number from 1 to 4.
6. Individually wrap the four assemblies in bubble wrap.
7. Package the four assemblies for shipping.
8. Ship Assemblies 1 and 2 to Advanced Technologies, Pasadena CA. Electron beam weld.
9. After welding, helium leak check Assemblies 1 and 2.
10. Section, inspect and photomicrograph Assembly 1.
11. Vacuum fire Assembly 2 at 1373K for four hours. Pressure should be less than 1×10^{-5} psi.
12. Helium leak check Assembly 2.
13. Section, inspect and photomicrograph Assembly 2.
14. Ship Assemblies 3 and 4 to Applied Energy, Winchester MA. Electron beam weld.
15. After welding, helium leak check Assemblies 3 and 4.
16. Section, inspect and photomicrograph Assembly 3.
17. Vacuum fire Assembly 4 at 1373K for four hours. Pressure should be less than 1×10^{-5} psi.
18. Helium leak check Assembly 4.
19. Section, inspect and photomicrograph Assembly 4.



Task 2

Unfired

Unplated Ud720LI tube.

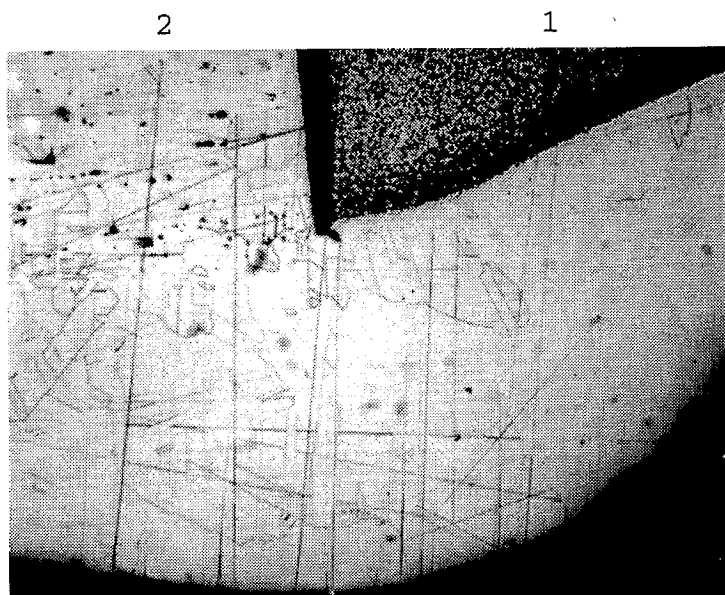
Unplated In718 plate.

Electron beam welded

Sample No. 1.

Magnification: 50x

Etched



Task 2

Vacuum fired at 1373K for 4 hr.

Unplated Ud720LI tube.

Unplated In718 plate.

Electron beam welded.

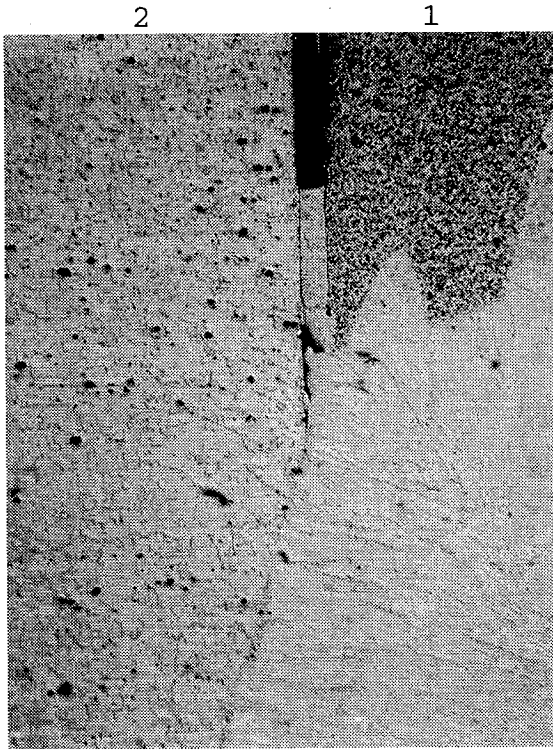
Sample No 2.

Magnification: 50x

Etched

1 Udimet 720LI
2 Inconel 718

Appendix K



Task 2

Unfired

Unplated Ud720LI tube.

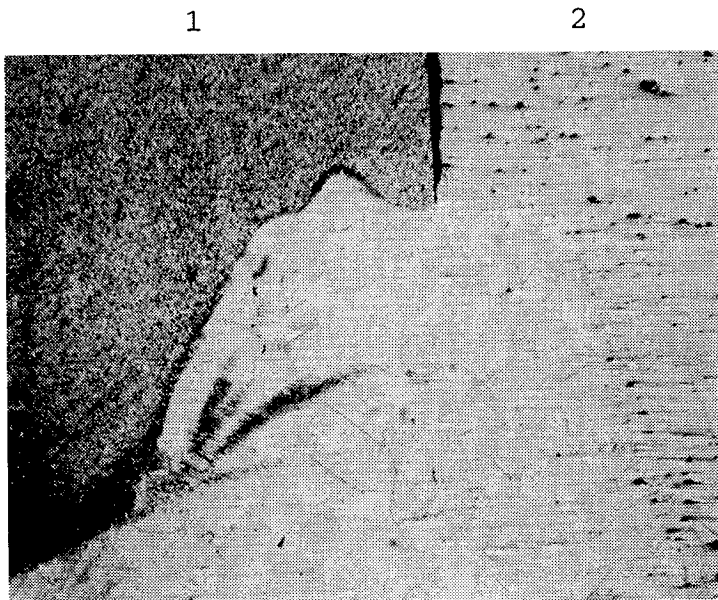
Unplated In718 plate.

Electron beam welded.

Sample No. 3

Magnification: 50x

Etched



Task 2

Vacuum fired at 1373K for 4 hr.

Unplated Ud720LI tube.

Unplated In718 plate.

Electron beam welded.

Sample No. 4

Magnification: 50x

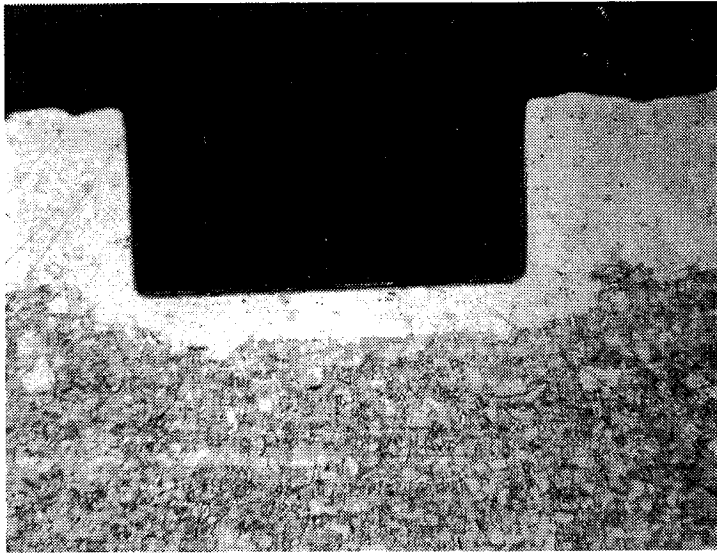
Etched

1 Udimet 720LI
2 Inconel 718

Appendix K

APPENDIX L

**PHOTOMICROGRAPHS FOR NICKEL ALUMINIDE COATED INCONEL 718 PARALLEL
GROOVE SPECIMENS COATED USING THE TWO-STEP
NICKEL ALUMINIDE APPLICATION PROCESS**



Task 3

Unfired

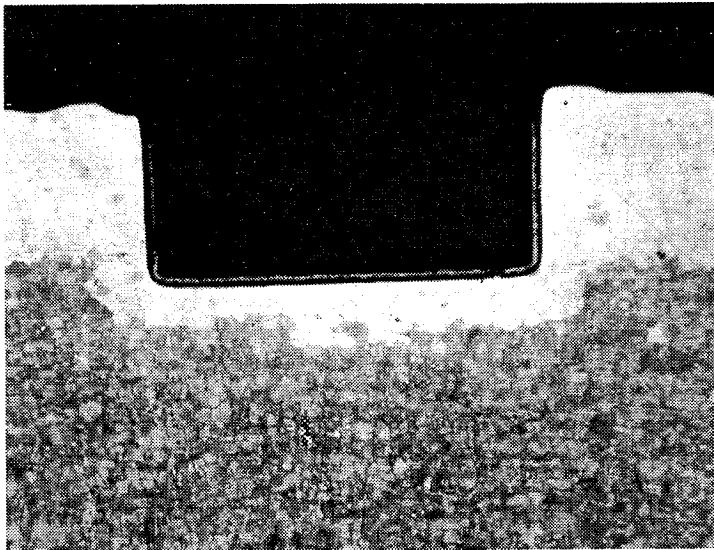
In718

2-step nickel aluminide coating.

Groove depth: 0.051cm.

Magnification: 50x

Etched



Task 3

Vacuum fired: 1073K for 8 hr.

In718

2-step nickel aluminide coating.

Groove depth: 0.051cm.

Magnification: 50x

Etched



Task 3

Vacuum fired: 1073K for 8 hr.

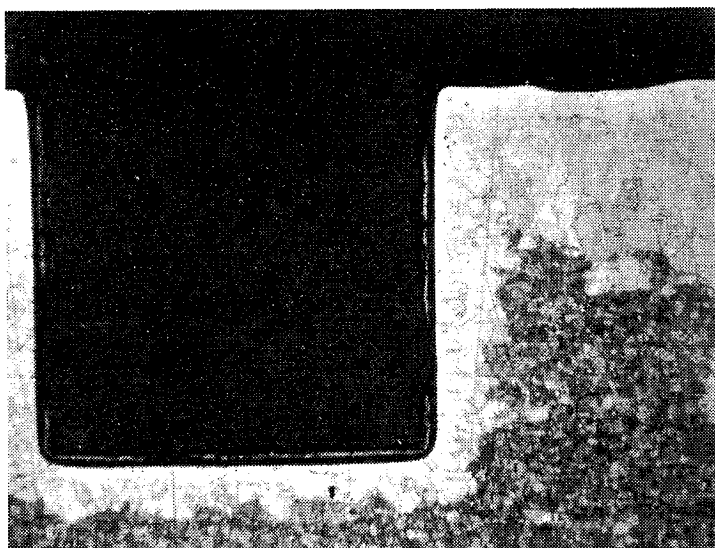
In718

2-step nickel aluminide coating.

Groove depth: 0.076cm.

Magnification: 50x

Etched



Task 3

Vacuum fired: 1073K for 8 hr.

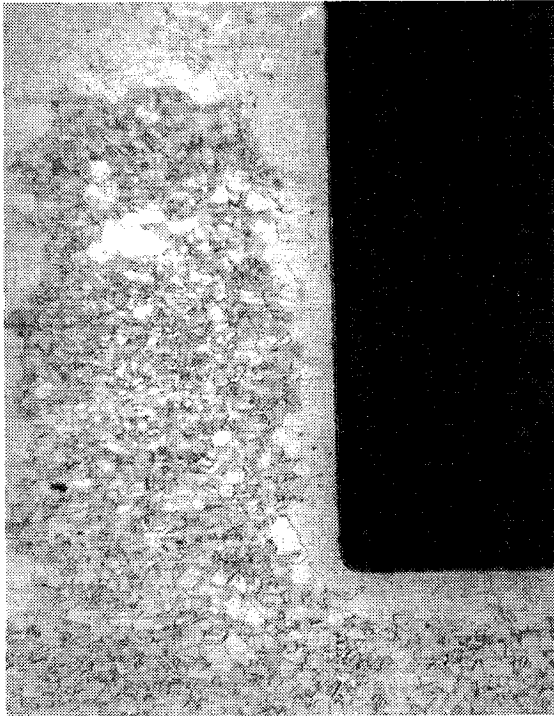
In718

2-step nickel aluminide coating.

Groove depth: 0.102cm.

Magnification: 50x

Etched



Task 3

Unfired

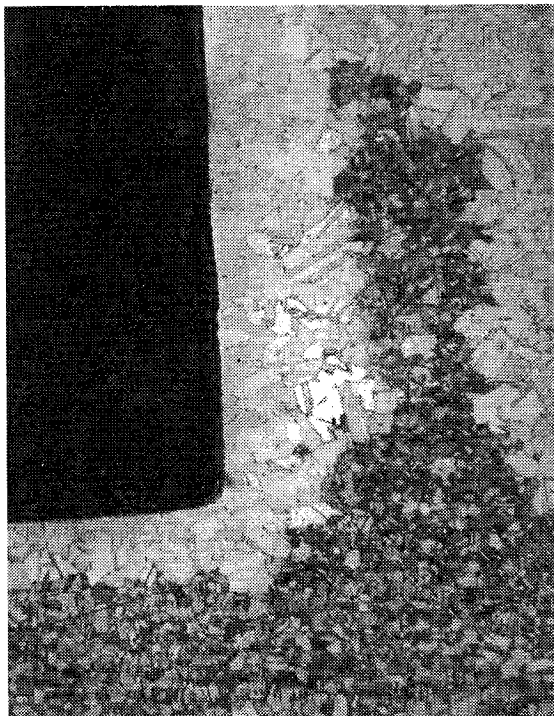
In718

2-step nickel aluminide coating.

Groove depth: 0.178cm.

Magnification: 50x

Etched



Task 3

Vacuum fired: 1073K for 8 hr.

In718

2-step nickel aluminide coating.

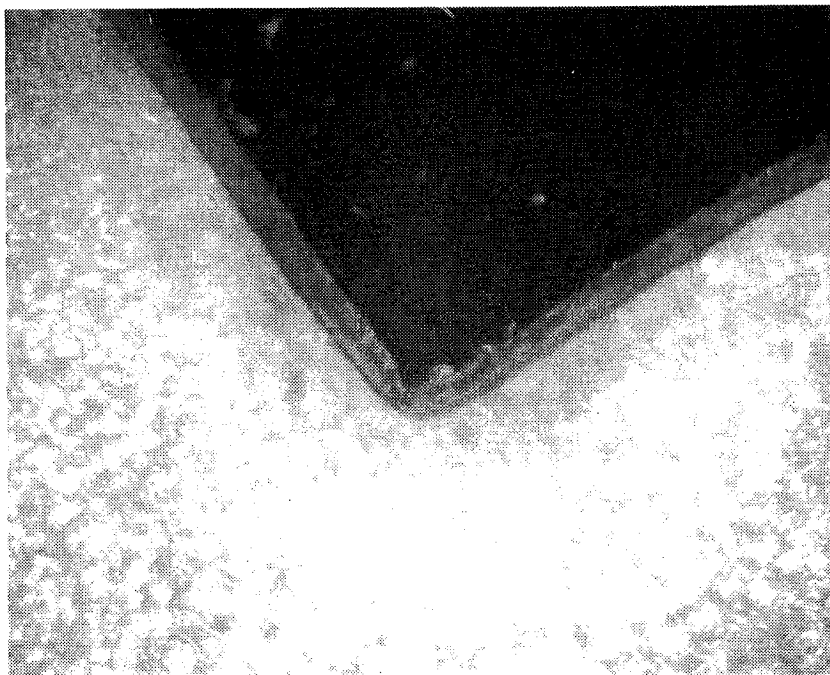
Groove depth: 0.178cm.

Magnification: 50x

Etched

APPENDIX M

PHOTOMICROGRAPHS FOR ALUMINUM DOPED SODIUM GROOVE PIPES NOS. 1 AND 4



Task 3
Sample No. 31

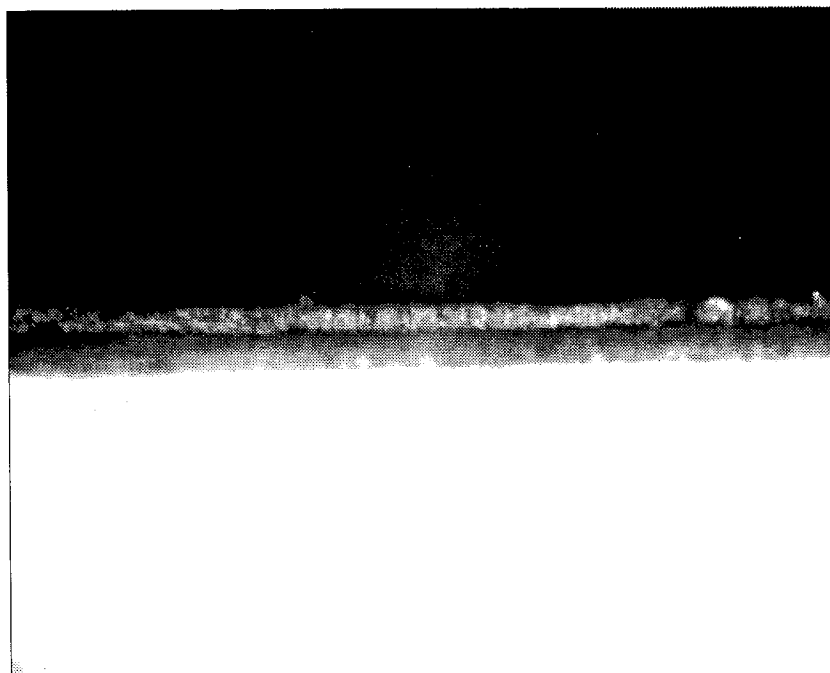
Groove Pipe No. 1
In718 groove specimen.
Groove depth: 0.051cm.

1.0in from fill tube end cap end.

913K for 1000 hours,
1RPM, horizontal

Sodium: 58g
Aluminum: 6g

Magnification: 200x
Etched



Task 3

Sample No. 34

Groove Pipe No. 1

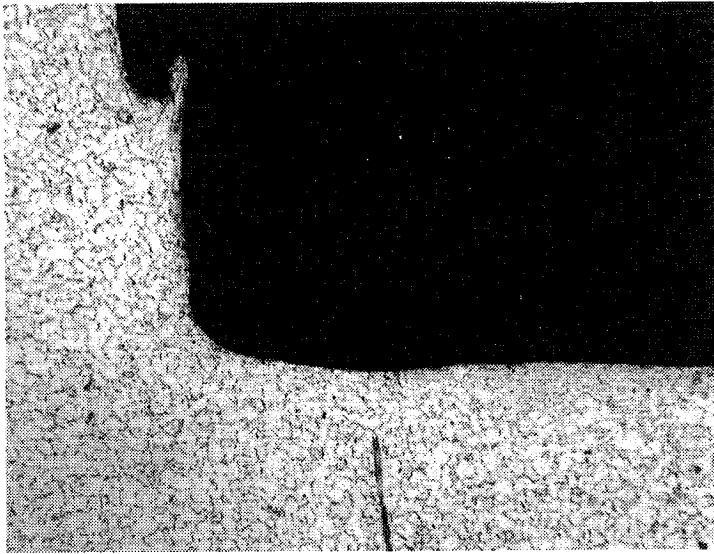
SS316 wall

1.0in from fill tube end cap.

913K for 1000 hours,
1RPM, horizontal

Sodium: 58g
Aluminum: 6g

Magnification: 200x
Etched



Task 3
Sample No. 65

Groove Pipe No. 4
In718 groove specimen.
Groove depth: 0.051cm.

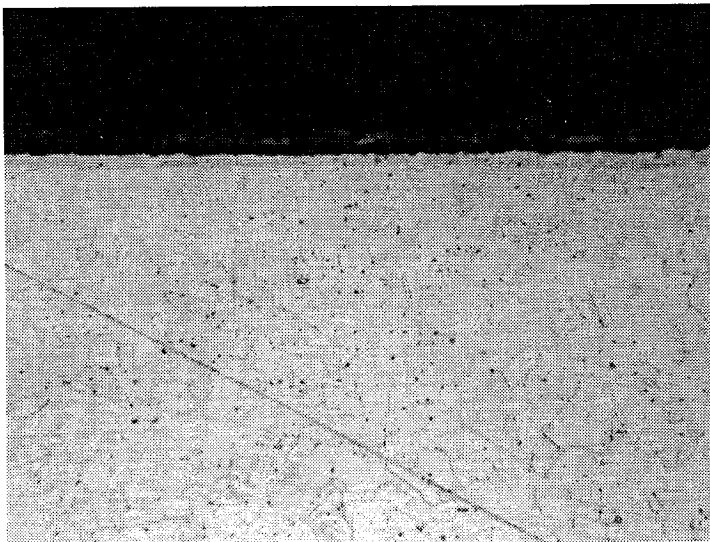
1.0in from fill tube end cap end.

913K for 1000 hours,
1RPM, horizontal

Sodium: 58g
Aluminum: 18g

Magnification: 200x
Etched

NOTE: Machining imperfection



Task 3

Sample No. 62

Groove Pipe No. 4

SS316 wall

1.0in from fill tube end cap.

913K for 1000 hours,
1RPM, horizontal

Sodium: 58g
Aluminum: 18g

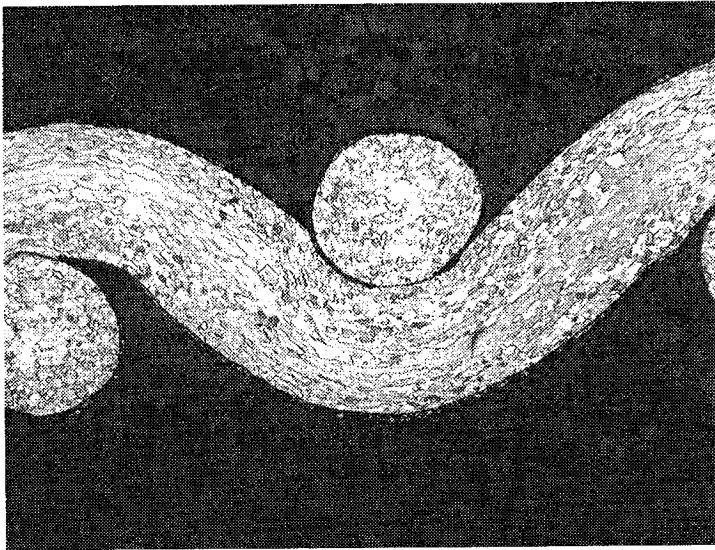
Magnification: 200x
Etched

APPENDIX N

PHOTOMICROGRAPHS FOR NICKEL ALUMINIDE COATED NICKEL 200

SCREEN SPECIMENS COATED USING THE ONE-STEP AND TWO-STEP

NICKEL ALUMINIDE APPLICATION PROCESS



Task 3

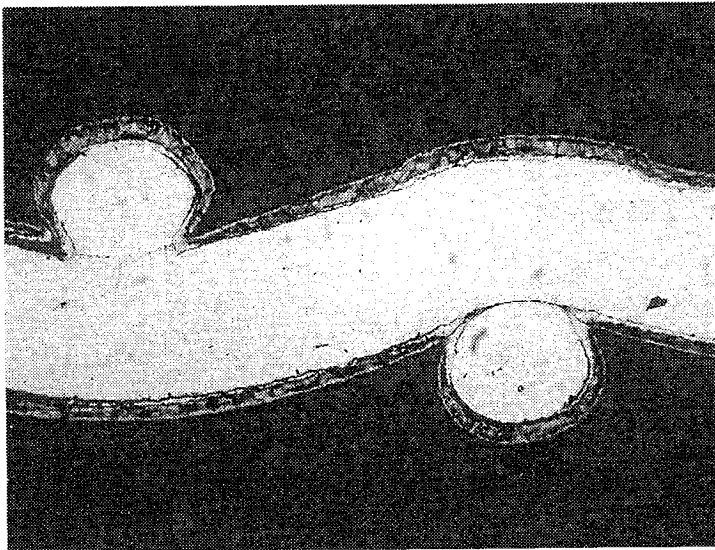
Unfired

100 mesh Ni200 screen.

1-step nickel aluminide coating.

Magnification: 200x

Etched



Task 3

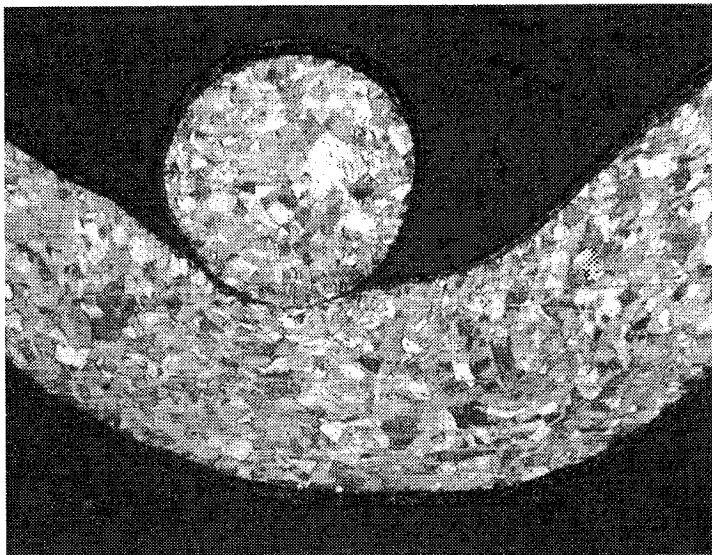
Vacuum fired: 1073K for 8 hr.

100 mesh Ni200 screen.

1-step nickel aluminide coating.

Magnification: 200x

Etched



Task 3

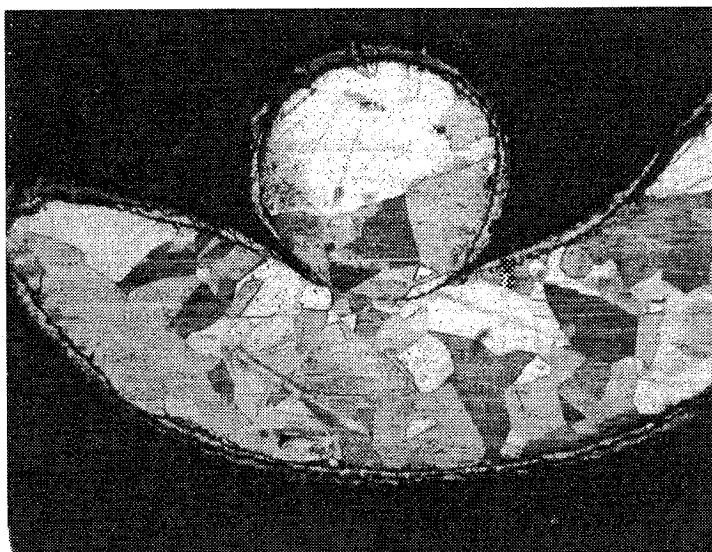
Unfired

60 mesh Ni200 screen.

1-step nickel aluminide coating.

Magnification: 200x

Etched



Task 3

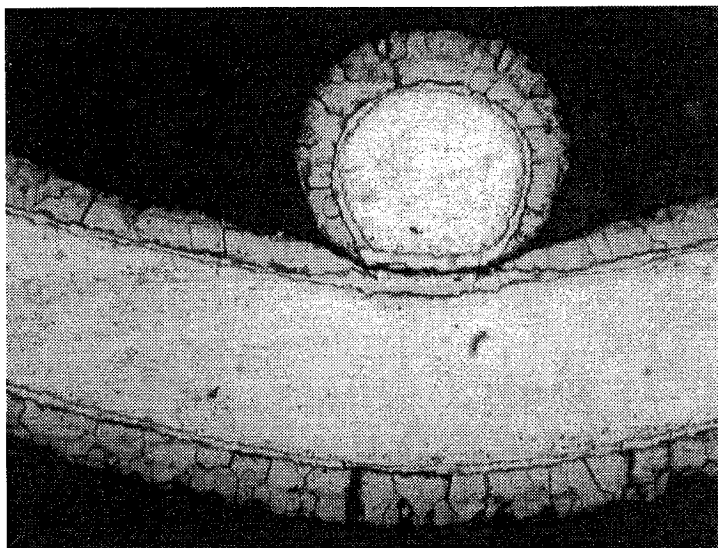
Vacuum fired: 1073K for 8 hr.

60 mesh Ni200 screen.

1-step nickel aluminide coating.

Magnification: 200x

Etched



Task 3

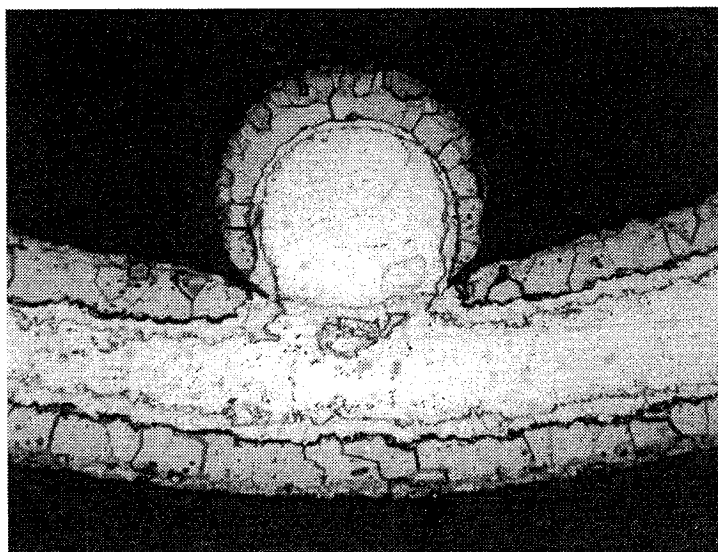
Unfired

60 mesh Ni200 screen.

2-step nickel aluminide coating.

Magnification: 200x

Etched



Task 3

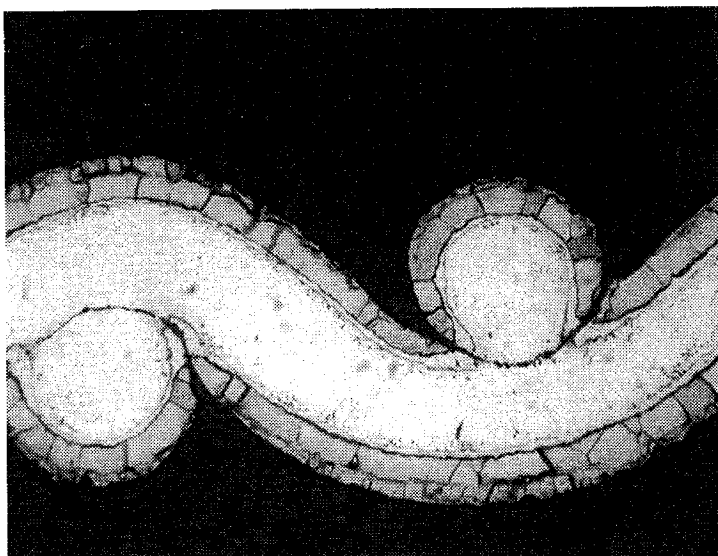
Vacuum fired: 1073K for 8 hr.

60 mesh Ni200 screen.

2-step nickel aluminide coating.

Magnification: 200x

Etched



Task 3

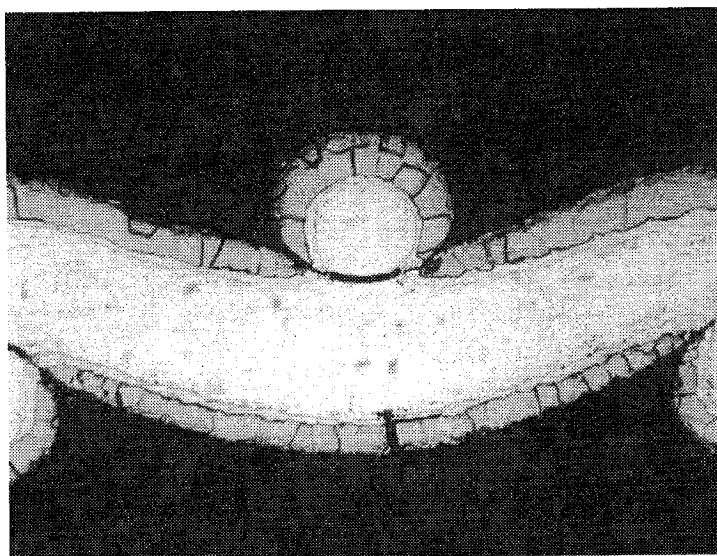
Unfired

100 mesh Ni200 screen.

2-step nickel aluminide coating.

Magnification: 200x

Etched



Task 3

Vacuum fired: 1073K for 8 hr.

100 mesh Ni200 screen.

2-step nickel aluminide coating.

Magnification: 200x

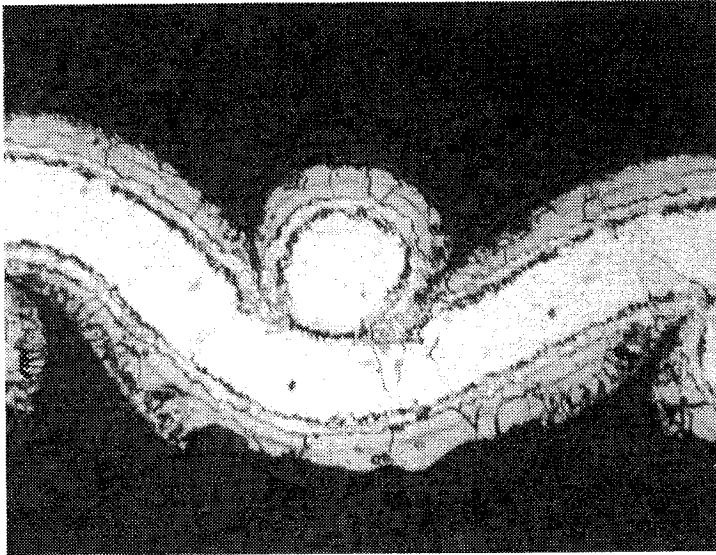
Etched

APPENDIX O

PHOTOMICROGRAPHS FOR UNCOATED NICKEL 200 SCREEN SPECIMENS

SPOT WELDED TO UNCOATED INCONEL 718 SUBSTRATES AND THEN COATED AS AN

ASSEMBLY WITH THE ONE-STEP NICKEL ALUMINIDE PROCESS



Task 3

Unfired

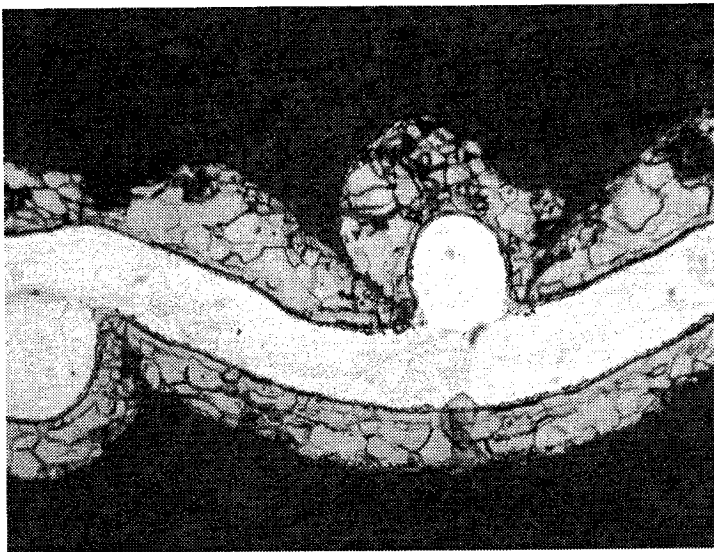
100 mesh Ni200 screen.

1-step nickel aluminide coating.

After spot welding.

Magnification: 200x

Etched



Task 3

Vacuum fired: 1073K for 8 hr.

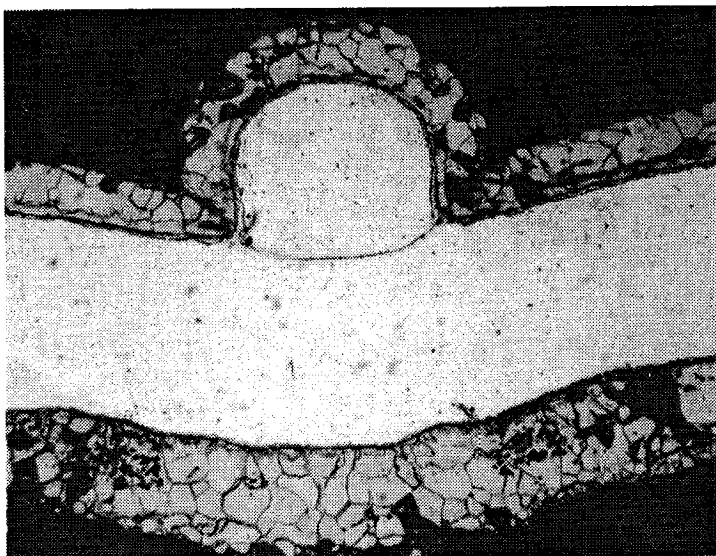
100 mesh Ni200 screen.

1-step nickel aluminide coating.

After spot welding.

Magnification: 200x

Etched



Task 3

Unfired

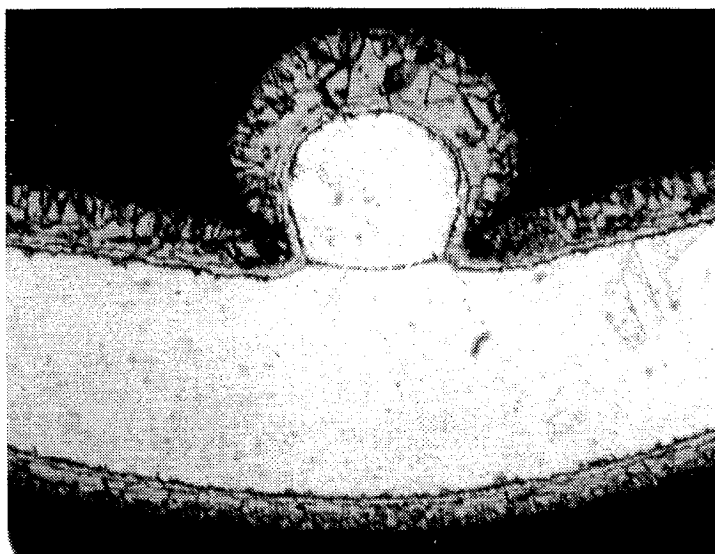
60 mesh Ni200 screen.

1-step nickel aluminide coating.

After spot welding.

Magnification: 200x

Etched



Task 3

Vacuum fired: 1073K for 8 hr.

60 mesh Ni200 screen.

1-step nickel aluminide coating.

After spot welding.

Magnification: 200x

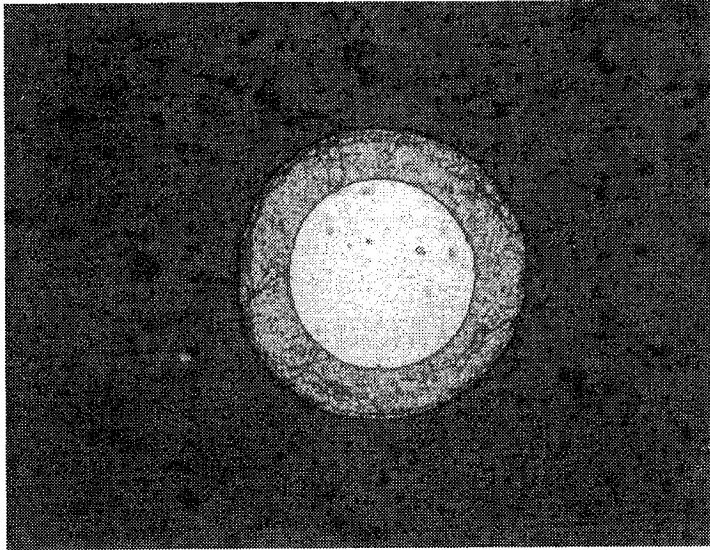
Etched

APPENDIX P

PHOTOMICROGRAPHS FOR NICKEL ALUMINIDE COATED 0.015cm DIAMETER

NICKEL 200 WIRE SPECIMENS COATED USING THE ONE-STEP

NICKEL ALUMINIDE APPLICATION PROCESS



Task 3

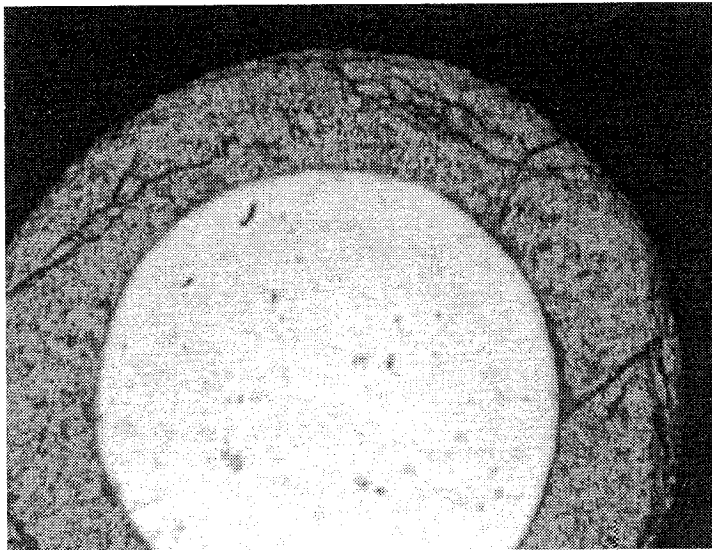
Unfired

0.015cm diameter Ni200 wire.

1-step nickel aluminide coating.

Magnification: 100x

Etched



Task 3

Vacuum fired: 1073K for 8 hr.

0.015cm diameter Ni200 wire.

1-step nickel aluminide coating.

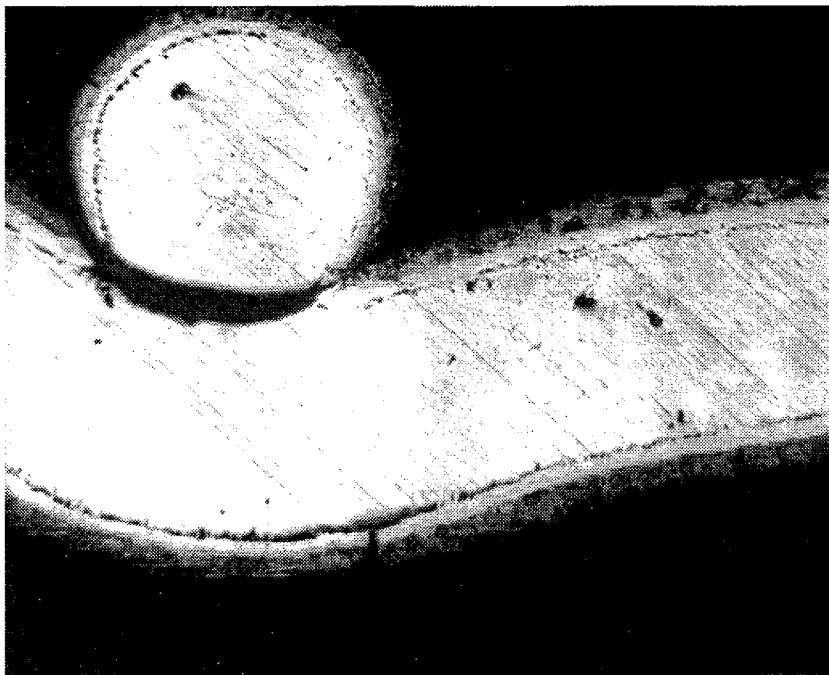
Magnification: 200x

Etched

APPENDIX Q

PHOTOMICROGRAPHS FOR THE TWO-STEP ALUMINUM TO NICKEL ALUMINIDE

COATED NICKEL 200 SCREEN SPECIMENS



Task 3

Sample No. 3

Vapor deposited aluminum.
Vacuum fired: 1293K for 2 hr.

60 mesh Ni200 screen.

2-step aluminum to nickel
aluminide coating.

Magnification: 250x

Etched



Task 3

Sample No. 4

Vapor deposited aluminum.
Vacuum fired: 1293K for 2 hr.

100 mesh Ni200 screen.

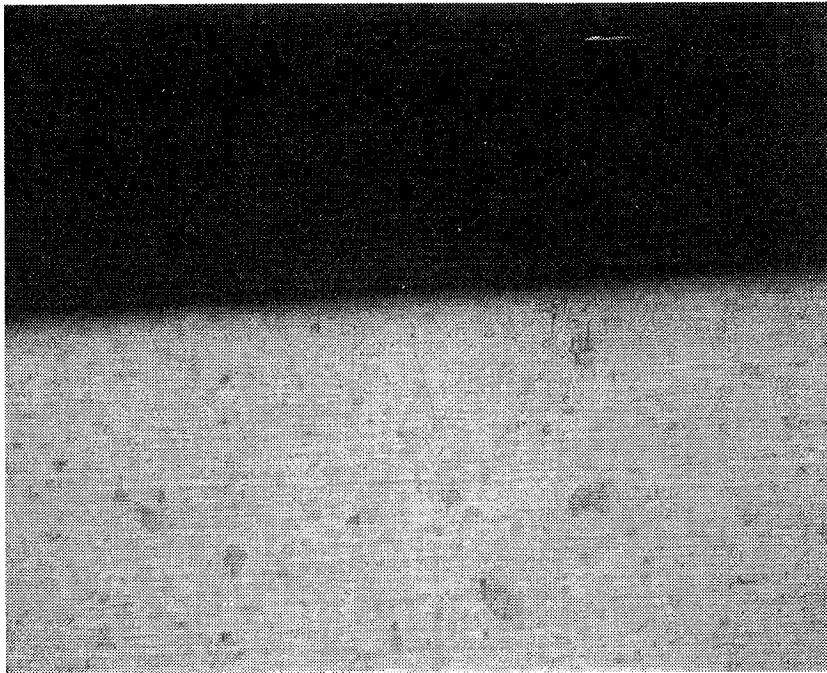
2-step aluminum to nickel
aluminide coating.

Magnification: 250x

Etched

APPENDIX R

PHOTOMICROGRAPHS FOR ALUMINUM DOPED SODIUM SCREEN PIPES NOS. 1 AND 3



Task 3

Sample No. 43

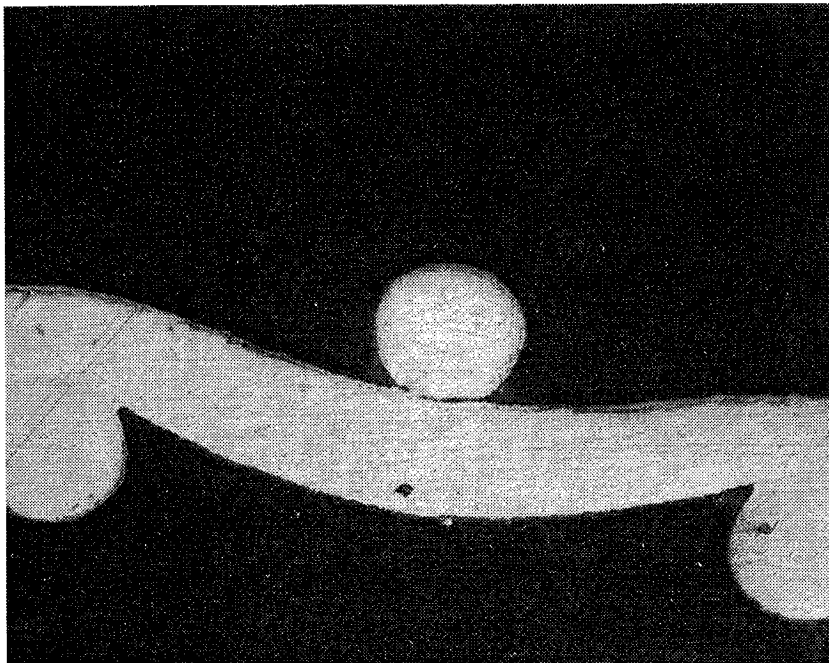
Screen Pipe No. 1
SS316 wall.
1.0in from fill tube end cap.

913K for 1000 hours,
1RPM, horizontal

Sodium: 58g
Aluminum: 6g

Magnification: 196x

Etched



Task 3

Sample No. 46

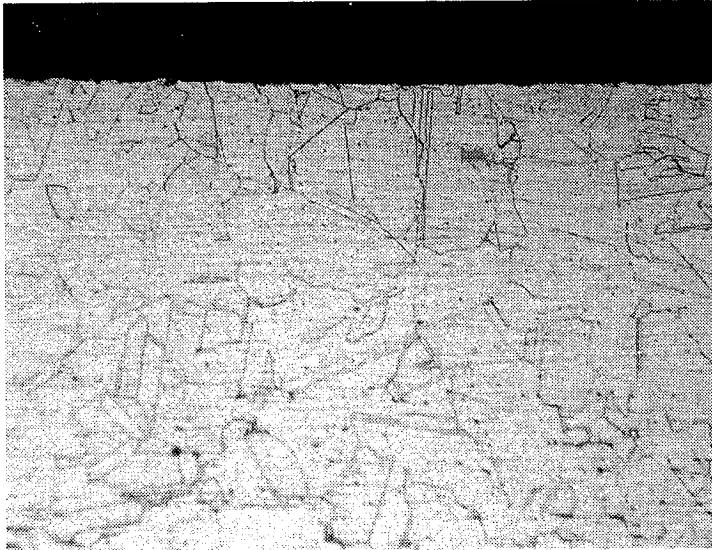
Screen Pipe No. 1
100 mesh Ni200 screen.
1.0in from fill tube end cap.

913K for 1000 hours,
1RPM, horizontal

Sodium: 58g
Aluminum: 6g

Magnification: 196x

Etched



Task 3

Sample No. 86

Screen Pipe No. 3

SS316 wall.

1.0in from fill tube end cap.

913K for 1000 hours,

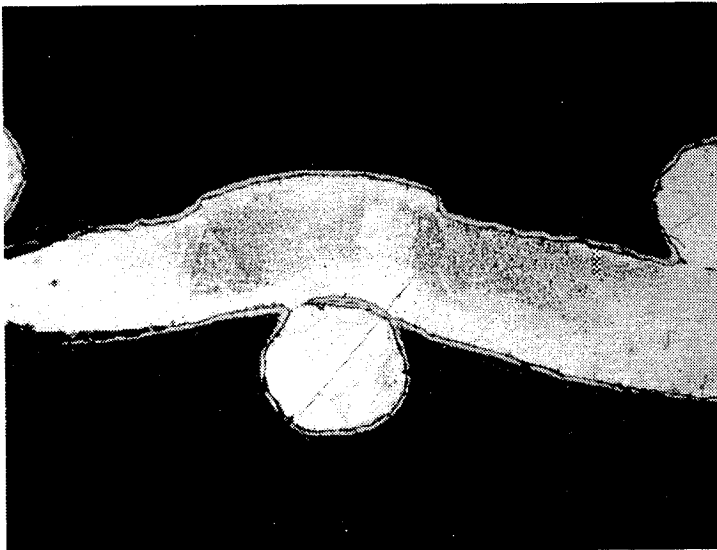
1RPM, horizontal

Sodium: 58g

Aluminum: 18g

Magnification: 200x

Etched



Task 3

Sample No. 89

Screen Pipe No. 3

100 mesh Ni200 screen.

1.0in from fill tube end cap.

913K for 1000 hours,

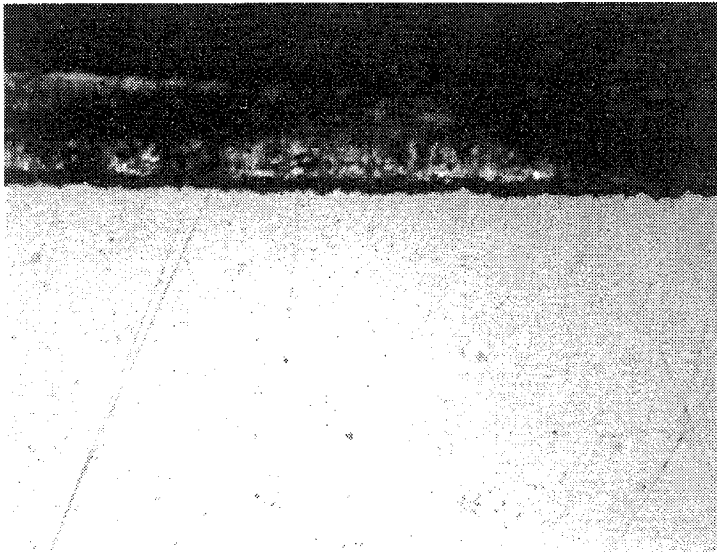
1RPM, horizontal

Sodium: 58g

Aluminum: 18g

Magnification: 200x

Etched



Task 3

Sample No. 88

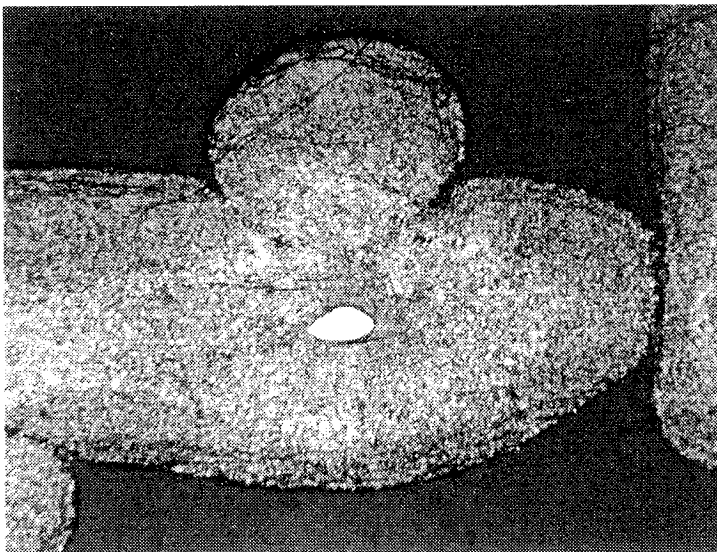
Screen Pipe No. 3
SS316 wall.
1.0in from solid tube end cap.

913K for 1000 hours,
1RPM, horizontal

Sodium: 58g
Aluminum: 12g

Magnification: 200x

Etched



Task 3

Sample No. 91

Screen Pipe No. 3
100 mesh Ni200 screen.
1.0in from solid tube end cap.

913K for 1000 hours,
1RPM, horizontal

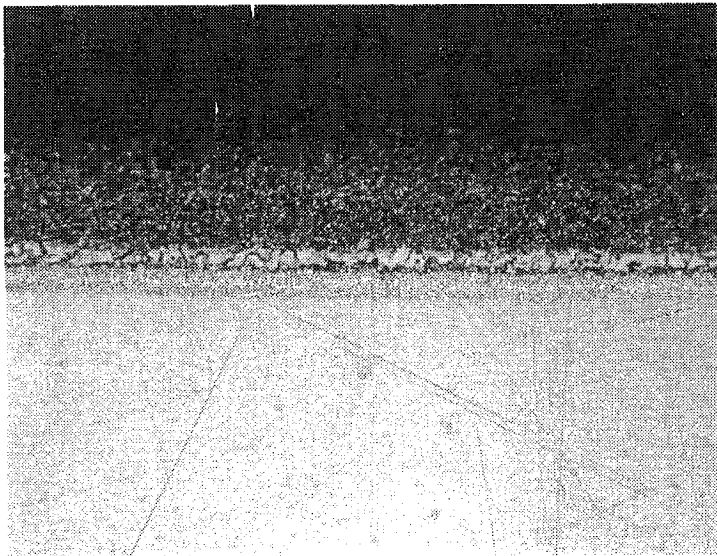
Sodium: 58g
Aluminum: 12g

Magnification: 200x

Etched

APPENDIX S

PHOTOMICROGRAPHS FOR TASK 4 PRE-MATRIX HEAT PIPES NOS. 1 - 3



Task 4
Sample No. 3

Pre-Matrix Pipe No. 1

In718 wall: 2-step nickel
aluminide coating.

Middle of condenser.

1073K for 1000 hours.
Condenser below evaporator.

Sodium: 58g

Magnification: 200x
Etched



Task 4
Sample No. 4

Pre-Matrix Pipe No. 1

100 mesh Ni200 screen.
1-step nickel aluminide coating.

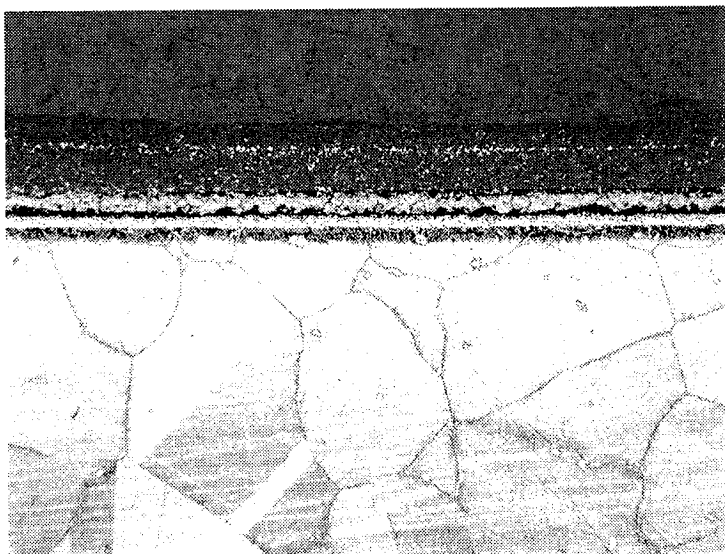
Middle of condenser.

1073K for 1000 hours.
Condenser below evaporator.

Sodium: 58g

Magnification: 100x

Etched



Task 4
Sample No. 7

Pre-Matrix Pipe No. 2

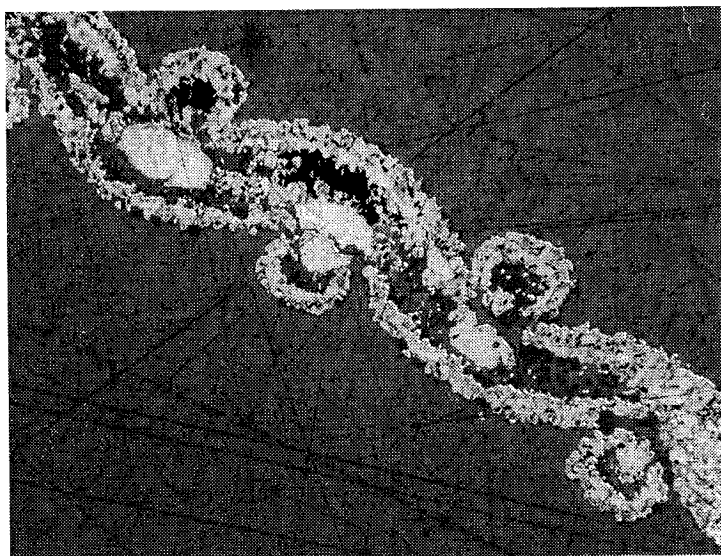
In718 wall: 2-step nickel
aluminide coating.

Middle of condenser.

1073K for 1000 hours.
Condenser below evaporator.

Sodium: 58g

Magnification: 200x
Etched



Task 4
Sample No. 8

Pre-Matrix Pipe No. 2

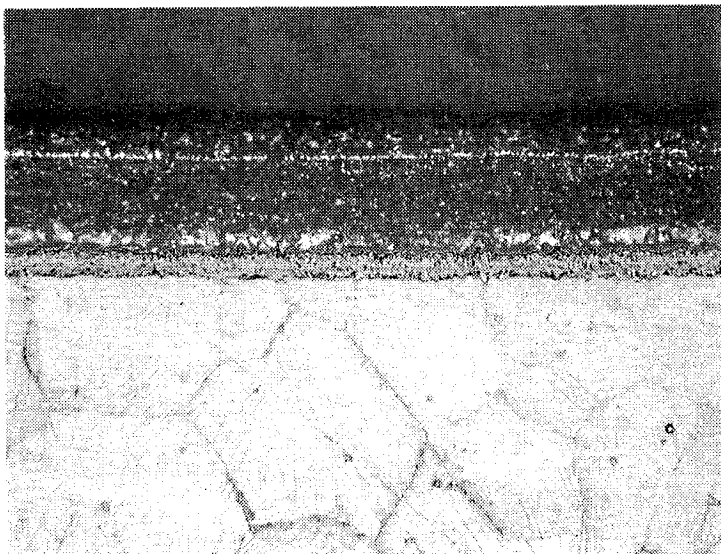
100 mesh Ni200 screen.
2-step aluminum-to-nickel
aluminide coating.

Middle of condenser.

1073K for 1000 hours.
Condenser below evaporator.

Sodium: 58g

Magnification: 100x
Etched



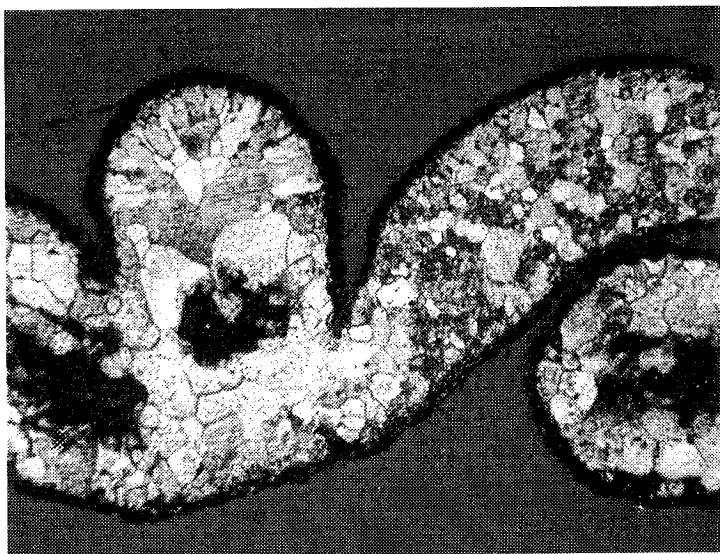
Task 4
Sample No. 11
Pre-Matrix Pipe No. 3

In718 wall: 2-step nickel
aluminide coating.
Middle of condenser.

913K for 1000 hours, 1RPM,
horizontal.
Sodium: 58g,
Aluminum: 18g

1073K for 1000 hours.
Condenser below evaporator.
Sodium: 58g

Magnification: 200x
Etched



Task 4
Sample No. 12
Pre-Matrix Pipe No. 3

Ni200 screen initially uncoated..
Middle of condenser.

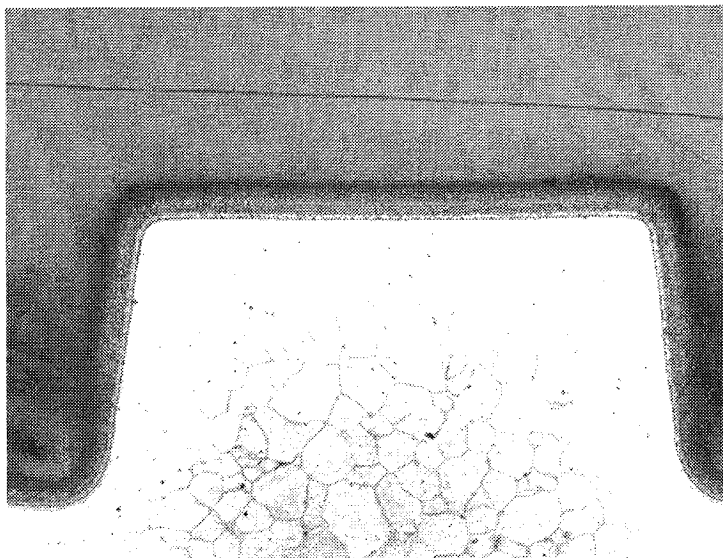
913K for 1000 hours, 1RPM,
horizontal.
Sodium: 58g,
Aluminum: 18g

1073K for 1000 hours.
Condenser below evaporator.
Sodium: 58g

Magnification: 200x
Etched

APPENDIX T
PHOTOMICROGRAPHS FOR DURABILITY HEAT PIPES NOS. 1 - 4

Appendix T



Task 4
Sample No. 15

Durability Pipe No. 1

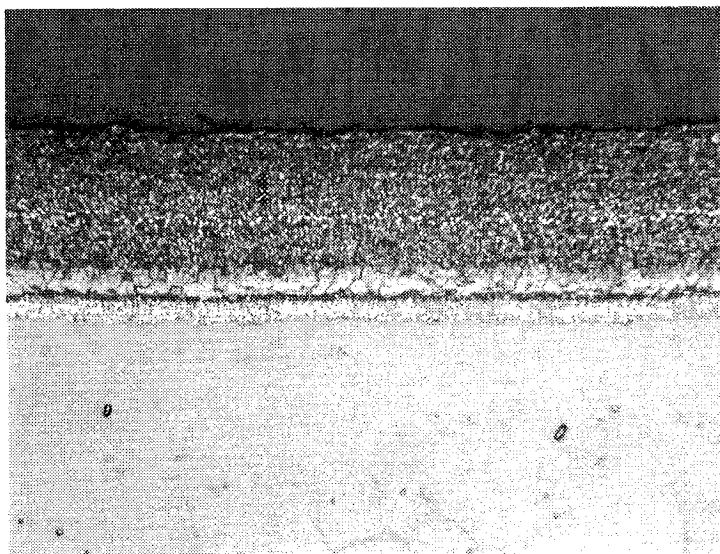
In718 wall / grooves.
2-step nickel aluminide coating.

Middle of condenser.

1073K for 1000 hours.
Condenser below evaporator.

Sodium: 58g

Magnification: 40x
Etched



Task 4

Sample No. 15

Durability Pipe No. 1

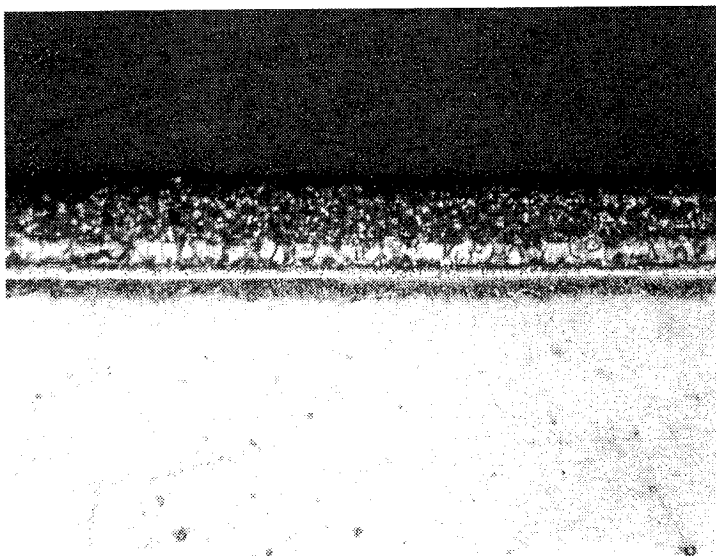
In718 wall / groove surfaces.
2-step nickel aluminide coating.

Middle of condenser.

1073K for 1000 hours.
Condenser below evaporator.

Sodium: 58g

Magnification: 200x
Etched



Task 4
Sample No. 19

Durability Pipe No. 2

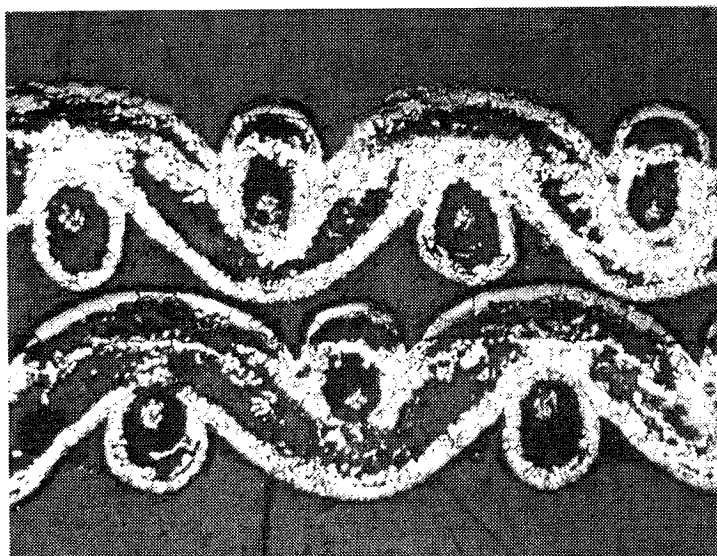
In718 wall: 2-step nickel
aluminide coating.

Middle of condenser.

1073K for 1000 hours.
Condenser below evaporator.

Sodium: 58g

Magnification: 200x
Etched



Task 4

Sample No. 20

Durability Pipe No. 2

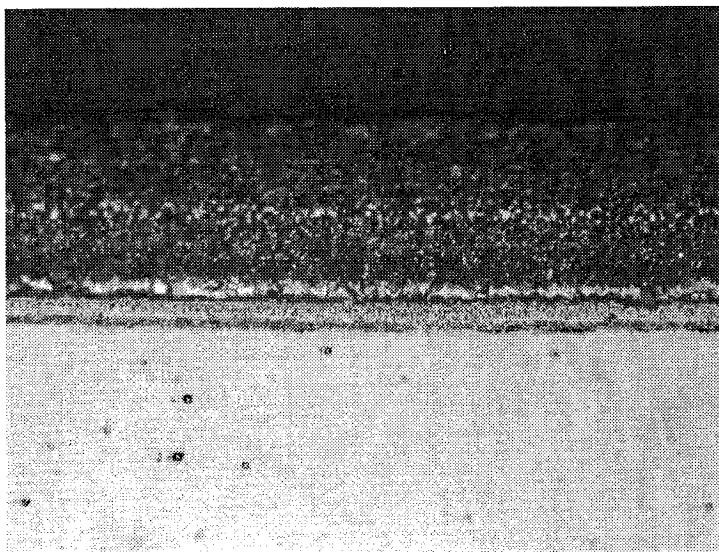
100 mesh Ni200 screen.
1-step nickel aluminide coating.

Middle of condenser.

1073K for 1000 hours.
Condenser below evaporator.

Sodium: 58g

Magnification: 100x
Etched



Task 4
Sample No. 25

Durability Pipe No. 3

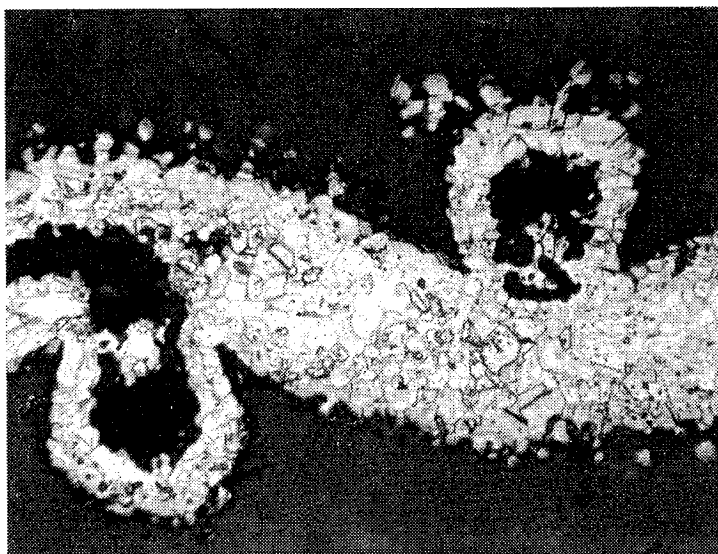
In718 wall: 2-step nickel
aluminide coating.

Middle of condenser.

1073K for 1000 hours.
Condenser below evaporator.

Sodium: 58g

Magnification: 200x
Etched



Task 4
Sample No. 26

Durability Pipe No. 3

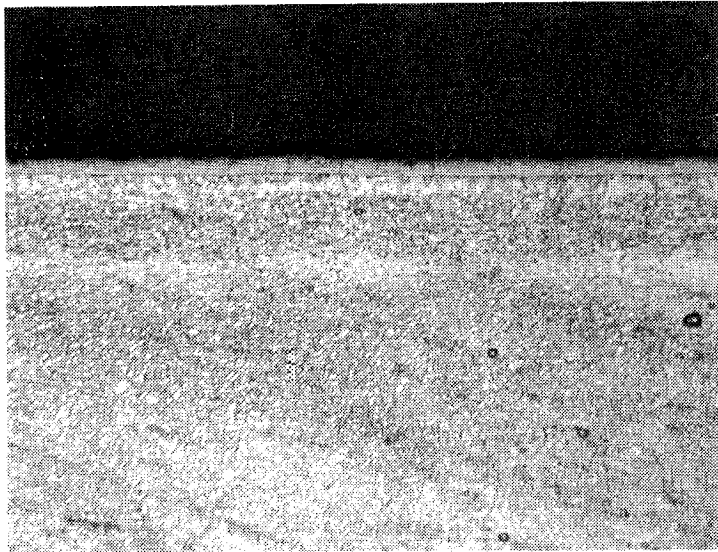
100 mesh Ni200 screen.
2-step aluminum-to-nickel
aluminide coating.

Middle of condenser.

1073K for 1000 hours.
Condenser below evaporator.

Sodium: 58g

Magnification: 200x
Etched



Task 4

Sample No. 29

Durability Pipe No. 4

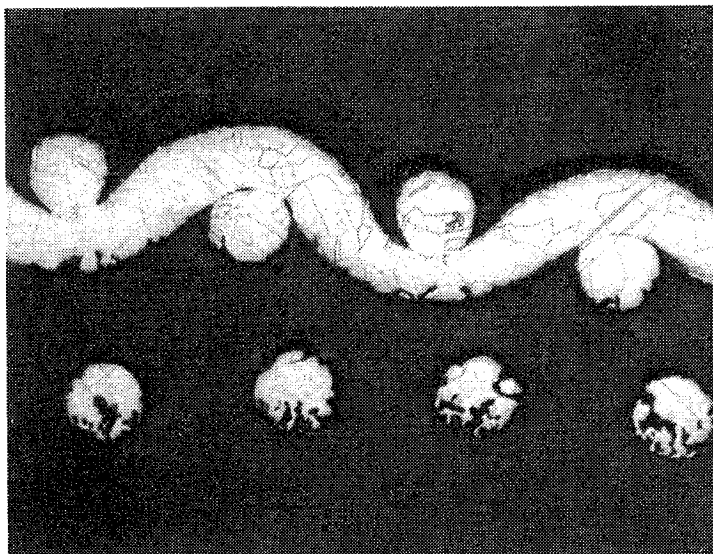
Uncoated In718 wall.

Middle of condenser.

1073K for 1000 hours.
Condenser below evaporator.

Sodium: 58g

Magnification: 200x
Etched



Task 4

Sample No. 30

Durability Pipe No. 4

Uncoated Ni200 screen.

Middle of condenser.

1073K for 1000 hours.
Condenser below evaporator.

Sodium: 58g

Magnification: 100x

Etched

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13. ABSTRACT (Maximum 200 words) The principal objective of this Phase II SBIR program was to develop and demonstrate a practically insoluble coating for nickel-based superalloys for Stirling engine heat pipe applications. Specific technical objectives of the program were (1) Determine the solubility corrosion rates for Nickel 200, Inconel 718, and Udimet 720LI in a simulated Stirling engine heat pipe environment, (2) Develop coating processes and techniques for capillary groove and screen wick structures, (3) Evaluate the durability and solubility corrosion rates for capillary groove and screen wick structures coated with an insoluble coating in cylindrical heat pipes operating under Stirling engine conditions, (4) Design and fabricate a coated full-scale, partial segment of the current Stirling engine heat pipe for the Stirling Space Power Converter program. The work effort successfully demonstrated a two-step nickel aluminide coating process for groove wick structures and interior wall surfaces in contact with liquid metals; demonstrated a one-step nickel aluminide coating process for nickel screen wick structures; and developed and demonstrated a two-step aluminum-to-nickel aluminide coating process for nickel screen wick structures. In addition, the full-scale, partial segment was fabricated and the interior surfaces and wick structures were coated. The heat pipe was charged with sodium, processed, and scheduled to be life tested for up to ten years as a Phase III effort.				
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